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Investigation of the Phase Stabilizing Effect of 4 % Potassium Fluoride on Ammonium Nitrate

Ref.: R&D # 5955A-CH-01
Purchase Order DAJA 45-89-M-0078
Final Report

Dr. Walter Engel

September 1989
D 7507 Pfinztal-Berghausen
W. Germany

CONTENTS

1. INTRODUCTION

Ammonium nitrate is an interesting oxidiser in the field of propellants and explosives. The substance is cheap and readily availbale. The drawbacks of the substance are caused by its polymorphic properties. It crystallizes at ambient pressure in 5 modifications. Some phase transitions take place at temperatures, which occur under normal storage conditions. These transitions are connected with volume changes that influence severely the properties of products containing ammonium nitrate.

Many attempts have been made to overcome these difficulties. One of the approaches consist of incorporating different ions into the lattice to change the phase properties. In this context potassium fluoride has been used.

The doped ammonium nitrate material must be checked to make sure that no undesired phase transitions and volume changes occur in the interesting temperature interval.
This is normally done by the methods of thermal analysis like DSC and DTA. These methods register thermal effects without delivering direct information on the lattice.

In the case of ammonium nitrate, however, these elaborated methods are not sufficient. A variety of phase transitions may occur, so that the phase behaviour cannot be understood, if the participating phases cannot be identified. This goal can be reached with diffraction measurements like x-ray and neutron diffraction.

A measuring system was built up in the past, which allows automatic measurements of series of diffraction patterns while cycling a sample stepwise through the interesting temperature ranges. The series contain informations about phase transition temperatures and the participating phases.

The system was already used to investigate the phase properties of ammonium nitrate samples, which were melted with 2 weight % of potassium fluoride. The final report of these investigations was completed and transmitted in july 1989.

The contract was extended by five series:

Series	Temperature Program (°C)
KF 280988 2% KF, humide	20/ 80/-70/ 80
KF 270988 4% KF, dry	20/ 80/-70/ 80
KF 290988 4% KF, dry	20/-70/ 80/-70
KF 150988 4% KF, humide	20/ 80/-70/ 80
KF 061088 4% KF, humide	20/-70/ 80/-70

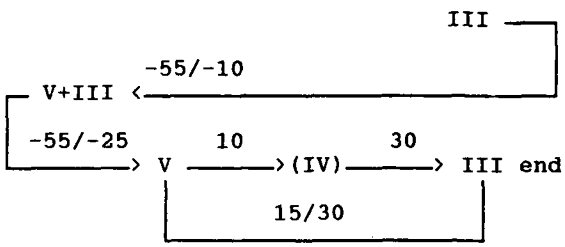
The series KF 280988 with 2% KF, humide, has been already reported in the final report about the investigations with samples containing 2% KF. The four series with samples containing 4% KF are included in this report.

The documentation of the four series has been organized by means of an appendix. It contains for each series (1) the original diffraction patterns, of which each second or third pattern has been plotted, (2) difference curves, the explanation of which is given in the appendix, (3) plots of the lattice distances d and (4) plots of the intensities of selected peaks.

2. RESULTS OF THE MEASUREMENTS

2.1 AMMONIUM NITRATE + 4% KF, DRY
TEMPERATURE PROGRAM 20/ 80/-70/ 80
Series KF 270988

At room temperature the sample consisted completely of phase III. The phase changes during the temperature program can be summarized as follows:



During the first heating cycle and the following cooling phase III remains the only phase until -10 °C, when phase V appears. On further cooling phase V increases until -55 °C, when the phase change III->V freezes, so that at -70 °C the peaks of phase III are still present.

During the second heating cycle the rest of phase III changes slowly into phase V. At -25 °C the peaks of phase III have practically disappeared phase V being the only phase. From +15 °C phase V decreases, changing into phase III and to a minor extent into phase IV, whose peaks disappear at 30 °C by changing into phase III.

The described changes can be seen in the original diffraction patterns in the appendix. The difference curves are not that much informative showing only the phase change V/III and V/IV during the second heating cycle.

The changes can also be followed by the decreasing and increasing intensities of the phase III and phase V peaks.

2.2 AMMONIUM NITRATE + 4% KF, DRY
TEMPERATURE PROGRAM 20/-70/ 80/-70
Series KF 290988

At room temperature the sample consisted completely of phase III. The phase changes during the temperature program can be summarized as follows:

```
                    -65/-45
 ┌── V+III   <──────────────── III
 │
 │-55/-20      5/25          35
 └──────>  V ──────> IV ───────> III ──┐
           │                      │    │
           │       15 / 35        │    │
           └──────────────────────┘    │
                                       │
                   -65/-50             │
  end V+III  <─────────────────────────┘
```

On cooling phase III remains the only stable phase down to -45°C, when the first peaks of phase V appear increasing its intensities until -65 °C. At this temperature the peaks of phase III still have the same intensity as those of phase V.

On heating phase V increases on the expense of phase III, the peaks of which remain visible with weak intensity. Slightly above 0 °C peaks of phase IV appear increasing up to 25 °C before they disappear at 35 °C due to the phase change IV/III. The phase changes V/IV/III, however, occur only in a small amount of the sample. The prevailing part changes directly from Phase V to III between 15 and 35 °C.

During the second cooling cycle the phase change III/V is observed between -50 and -65 °C, which agrees with the fuirst cooling cycle.

The phase changes can be followed in the diffraction patterns and with the intensities of selected peaks of the phases III, IV and V, which are found in the appendix. The difference curves are not so informative. They show only the transitions V/II and V/IV during the heating cycle.

2.3 AMMONIUM NITRATE + 4% KF, HUMIDE
TEMPERATURE PROGRAM 20/ 80/-70/ 80
Series KF 150988

At room temperature the sample consisted completely of phase III. The phase changes during the temperature program can be summarized as follows:

```
                                    III ──┐
         -10/-5                           │
 ┌── V  <─────────────────────────────────┘
 │
 │         0/+5          30
 └─> V  ─────────> IV ─────────> III end
```

On heating no phase change occurs. On cooling the whole sample changes in a narrow temperature interval from III to V. Few weak phase IV peaks indicate that a very small part changes from phase III to phase IV. During the following heating cycle the whole sample changes at 0-5 °C into phase IV. The transition IV/III follows again in a narrow temperature interval.

In the diffraction patterns the sample shows strong texture effects, which can be observed with the intensities of the (210) and (021)-Peaks of phase III. Normally these peaks have comparable intensity. In this case, however, the intensity of the (021)-peak is abnormally low. In the same way the (020)-peak of phase IV is not visible after the transition V/IV.

The difference curves show clearly the transition III/V on cooling and the transition IV/III on heating. The transiton V/IV is also visible. The d-curves of the lattice plane distances and the intensity curves are consistent with the described transition path.

2.4 AMMONIUM NITRATE + 4% KF, HUMIDE
TEMPERATURE PROGRAM 20/-70/ 80/-70
Series KF 061088

At room temperature the sample consisted completely of phase III. The phase changes during the temperature program can be summarized as follows:

```
            -40/-30           -10/+5
  ┌── V  <───────────  IV  <───────────  III
  │                    V
  │         -10/+5             20/30
  └───────────────────> IV  ───────────>  III ──┐
                                                │
                   -20/-5                       │
 end V (+III)  <────────────────────────────────┘
```

On cooling, phase III changes between +5 and -10 °C into the phases IV and V the peaks of both phases having the same intensity, before phase IV changes into phase V between -30 and -40 °C.

On heating the transitions V/IV and IV/III occur completely in rather narrow temperature intervals. Weak peaks of phase III become already visible at about 15 °C so that the results seem to be similar to the results with the dry samples.

During the second cooling cycle phase III changes directly into phase V. At -70 °C weak peaks of phase III are still visible. Possibly the sample has a lower water content due to a drying effect during the preceding heating.

In the diffraction patterns the (021)-peak of phase has again a reduced intensity. The described transitions are well visible. The difference curve DY shows also nicely the observed transitions.

3. DISCUSSION

The investigations were made to determine the influence of the added 4% KF on the phase properties of the material. The results show that the samples consist at room temperature totally of phase III. The material distinguishes itself clearly from samples with 2% KF, where phase IV was always present to a certain extent in the samples.

The phase changes of the different samples during the temperature programs show also essential differences to pure ammonium nitrate and to the samples with 2% KF. For a discussion the results were summarized in table 1.

It can be seen that at temperatures from 30-80 °C phase III is the only stable phase. On cooling, phase III is the only phase to temperatures below 0 °C. The results differ between the dry and the humide samples. With the dry samples, phase III changes into phase V not earlier than between -10 and -65 °C and its peaks are still distinctly visible at -70 °C.

With the humide samples the phase change V/III occurs at higher temperatures between 5 and -10 °C. Besides, the phase change is complete at -10 °C leaving no peaks of phase III. Another difference occurs in the series 0601088, when the phase change III/V is accompanied by the phase change III/IV.

During the heating cycles from -70 to 80 °C phase V changes in a different transition path to phase IV at about 0 °C, which is also observed with pure Ammonium nitrate and with ammonium nitrate + 2% KF. The transition, however, is frequently accompanied by the transition V/III. This is especially true for the dry samples.

As a result, the phase stabilisation of phase III has almost been reached with 4% KF. On cooling phase III is stable down to temperatures below 0 °C and especially in the dry samples down to temperatures of about -50 °C. The goal, however, is not yet completely reached on heating, when phase IV still occurs.

The differences between the dry and the humide samples allow some conclusions about the kinetic of the phase changes. As can be seen in table 1, the phase change III/V on cooling occurs in all the dry samples at lower temperatures than in the humide samples. This should be due to a retarded nucleation, which starts the phase change at temperatures, where equilibrium freezes, so that peaks of phase III are still visible at -70 °C changing into phase V on heating as soon as the equilibrium is no longer frozen.

In the humide samples an earlier nucleation allows a complete transition III/V at temperatures, when the equilibrium is not yet frozen.

The comparison of dry and humide samples with 2% KF reported in the previous report yields similar differences. Dry samples show a more complicated behaviour on cooling, when the phase changes III/IV and III/V occur simultaneously. With the humide sample the transition path is simplified in the same way as in humide samples with 4% KF.

4. SUMMARY

Samples of ammonium nitrate containing 4 weight % KF were investigated during cycling the material between -70 and 80 °C. The phases changes were observed by diffraction measurements over the whole temperature programs.

The incorporation of 4% KF into the ammonium nitrate lattice extends the stability range of phase III to deeper temperatures. The results differ in dry and humide samples. In dry samples phase III changes directly into phase V on cooling at temperatures between -10 and -65 °C. The transitions are not complete. Phase IV is excluded.

In the humide samples the transition into phase V is complete occuring at higher temperatures between 5 and -10 °C. Besides, phase IV cannot be excluded reliably.

The differences can be explained by a retarded nucleation of phase V in the dry samples.

On heating the transition V/IV occurs in all samples at temperatures slightly above 0 °C followed by the transition IV/III at 30-35 °C. Due to the extended stability range of phase III a direct transition V/III is observed in the dry samples.

Table 1: Summarized Phase Changes

Sample Temp.Program			P H A S E C H A N G E S									
			V<-III	V<-IV		V->IV		IV<-III	V->III	IV->III		
KF 270988 20/80/-70/80 dry										start	III	
		V+III	-55/-10								III	
		V+III	-55/-25		V				15/30		III	
					V	10	IV			30	III	end
KF 290988 20/-70/80/-70 dry		V+III	-65/-45								III	start
		V+III	-55/-20		V	5/25	IV			35	III	
					V				15/35		III	
	end	V+III	-65/-50								III	
KF 150988 20/80/-70/80 humide										start	III	
		V	-10/-5								III	
		V				0/+5	IV			30	III	end
KF 061088 20/-70/80/-70 humide		V		-40/-30			IV	-10/+5			III	start
		V	-10/+5								III	
		V				-10/+5	IV			20/30	III	
	end	V(+III)	-20/-5								III	

5. APPENDIX

A Peak List

B Experimental Details

C Concept of Difference Diagrams

D Series KF 270988

E Series KF 290988

F Series KF 150988

G Series KF 061088

APPENDIX A

PEAK LIST

Diffraction Angles of Ammonium Nitrate
Phases I, II, III, IV, V
with Chromium Radiation, Lamda=2.2896 A

Peak No.	2*Theta	(h k l) of PHASE I	II	III	IV	V
1	26,8				(001)IV	
	27,05		(001)II			(002)V
2	29,2			(101)III		
3	30,5	(100)I				
4	33,0		(110)II			(020)V
5	33,67					(200)V
6	33,8				(110)IV	
7	34,24			(111)III		
8	39,53			(120)III		
9	41,43			(210)III		
10	41,78			(021)III		
11	43,5		(111)II			(022)V
12	43,8	(110)I			(111)IV	
13	45,7			(121)III		
14	46,9				(200)IV	
15	46,9					(212)V
16	47,72		(200)II			(220)V
17	47,72			(211)III		
18	49,8					(221)V
19	50-51				(020)IV	
20	51,85			(220)III		
21	53,2		(210)II	(112)III		
22	53,8					(130)V
23	55,0				(201)IV	
24	55,7		(002)II			(004)V
25	56,54			(130)III		
26	57,4				(021)IV	
27	58,5					(014)V
28	59,0			(022)III		
29	60,4			(310)III		
30	60,9		(211)II	(202)III		
31	60,9				(221)IV	
32	61,2					(132)V
	61,2					(312)V
33	61,8				(012)IV	

APPENDIX B

EXPERIMENTAL DETAILS

X-ray radiation: chromium tube, monochromatic, primary monochromator

Method : angle dispersive

Detector : Position sensitive proportional counter (PSPD)

Angular speed : 10 °/min

Series	Temp. Program	Temp. interval	Angular Speed	No. of patterns
KF 270988 dried (0,15%)	20/ 80/-70/ 80	5	10	72
KF 290988 dried (0,18%)	20/-70/ 80/-70	5	10	78
KF 150988 (2,75% H2O)	20/ 80/-70/ 80	5	10	73
KF 061088 (2,75% H2O)	20/-70/ 80/-70	5	10	79

Ammonium nitrate was melted together with 4 weight % of KF. One part of the material was dried under vacuum at 65 °C for 5 days. The other part was stored for 5 hours at 23 °C and a humidity of 65%.

APPENDIX C

Concept of Difference Diagrams

The measuring system delivers a huge amount of data, which must be efficiently evaluated. A concept of difference diagrams was developed, which detects differences in the diagrams of a series without requiring much computing. The concept was published in J.Appl.Cryst.(1983) 16, 259-263.

The principle can be seen on the following page. If differences are formed between all corresponding channels of two patterns, a difference diagram is formed. A peak, which is present in both patterns with a slight difference of the position creates a double peak, half of with represents the positive and the other half the negative values.

If the difference is formed between two peaks with the same position, but with different intensity, the resulting difference peak has its values either in the negative or positive part.

When the absolute values of the difference formation are summed up, the sum correlates with the difference between the two patterns. If the sums between all the neighboured patterns are plotted against temperature, a curve results, which corresponds to a DSC curve (1).

When the difference formation occurs constantly between the first and all the other patterns, the corresponding curve has some similarity with a thermogravimetric curve (2).

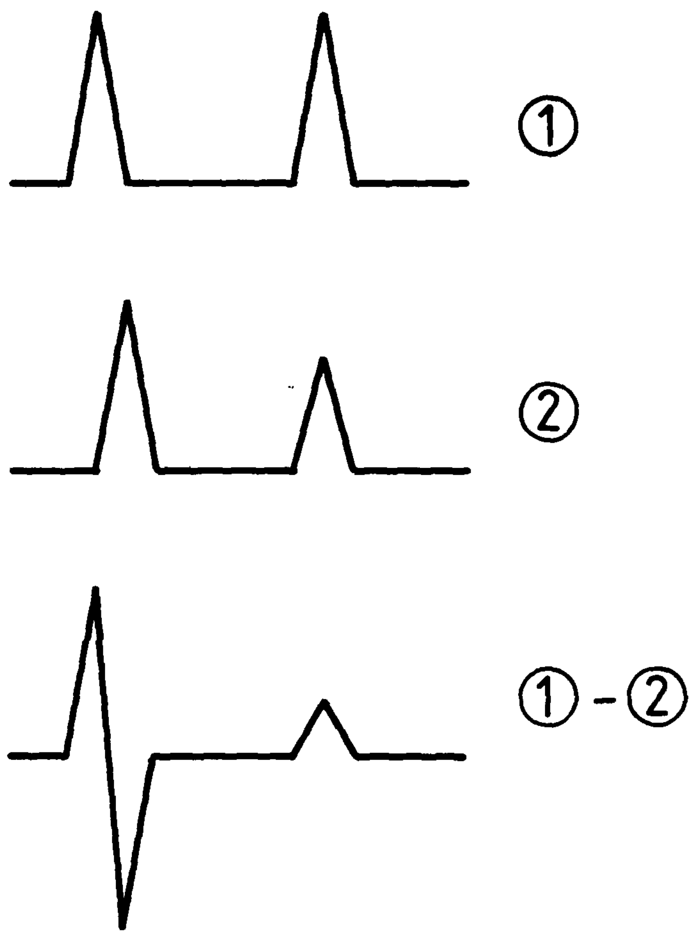
$$DY(T_i) = \sum_{j=1}^{N} |X_j(T_i) - X_j(T_{i-1})| \quad ; \; i = 2, \ldots, M \qquad (1)$$

$$Y(T_i) = \sum_{j=1}^{N} |X_j(T_i) - X_j(T_1)| \quad ; \; i = 2, \ldots, M \qquad (2)$$

X_j is the content of the j-th channel of the i-th pattern and N is the number of channels of the pattern. T_i represents the independent variable with which the i-th pattern was recorded and M is the number of patterns in a series.

The different curves are both calculated and plotted, as some effects as phase transitions show more distictly in the DY-curve, while thermal expansion can be seen only in th Y-curve.

Concept of difference diagrams

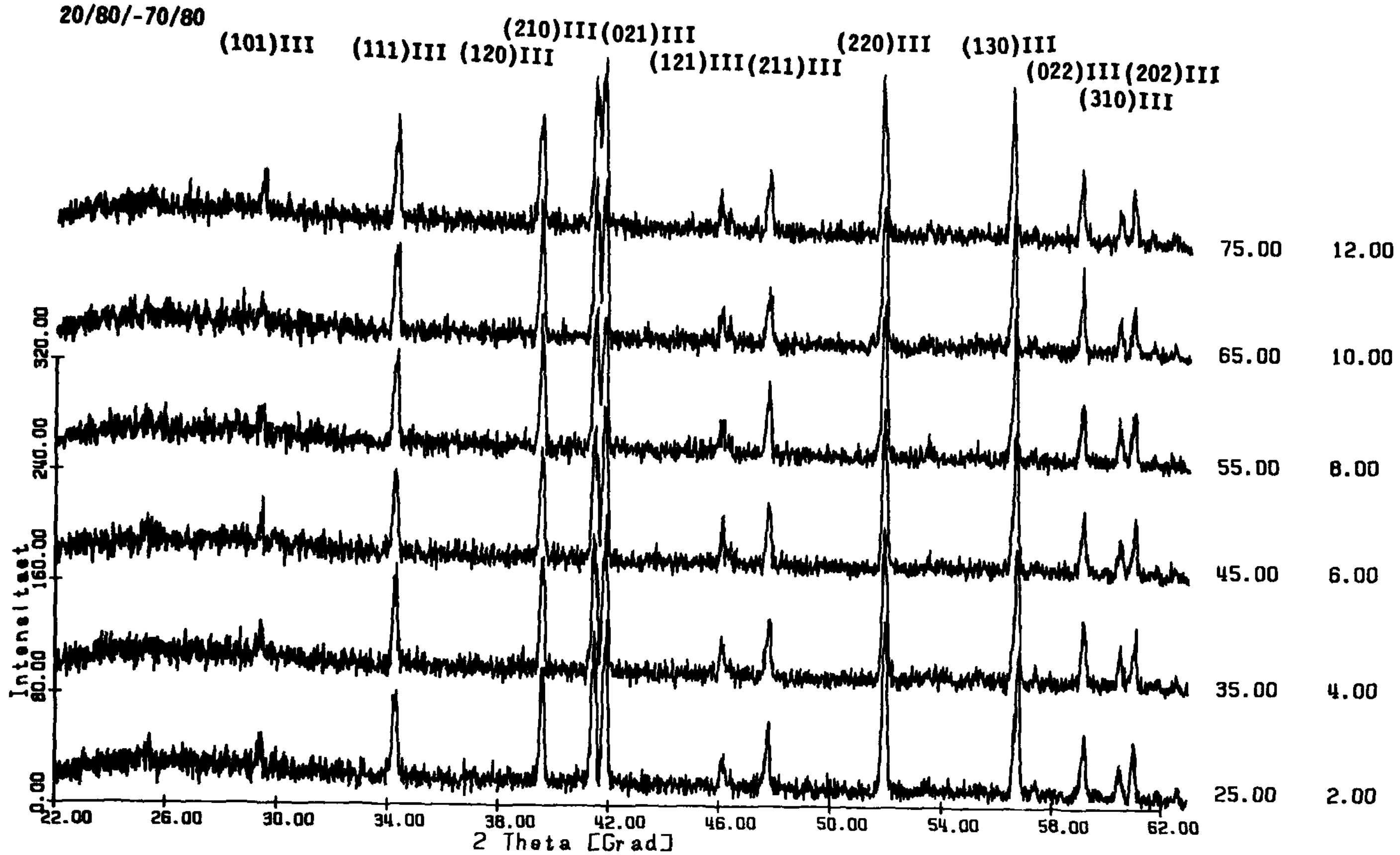


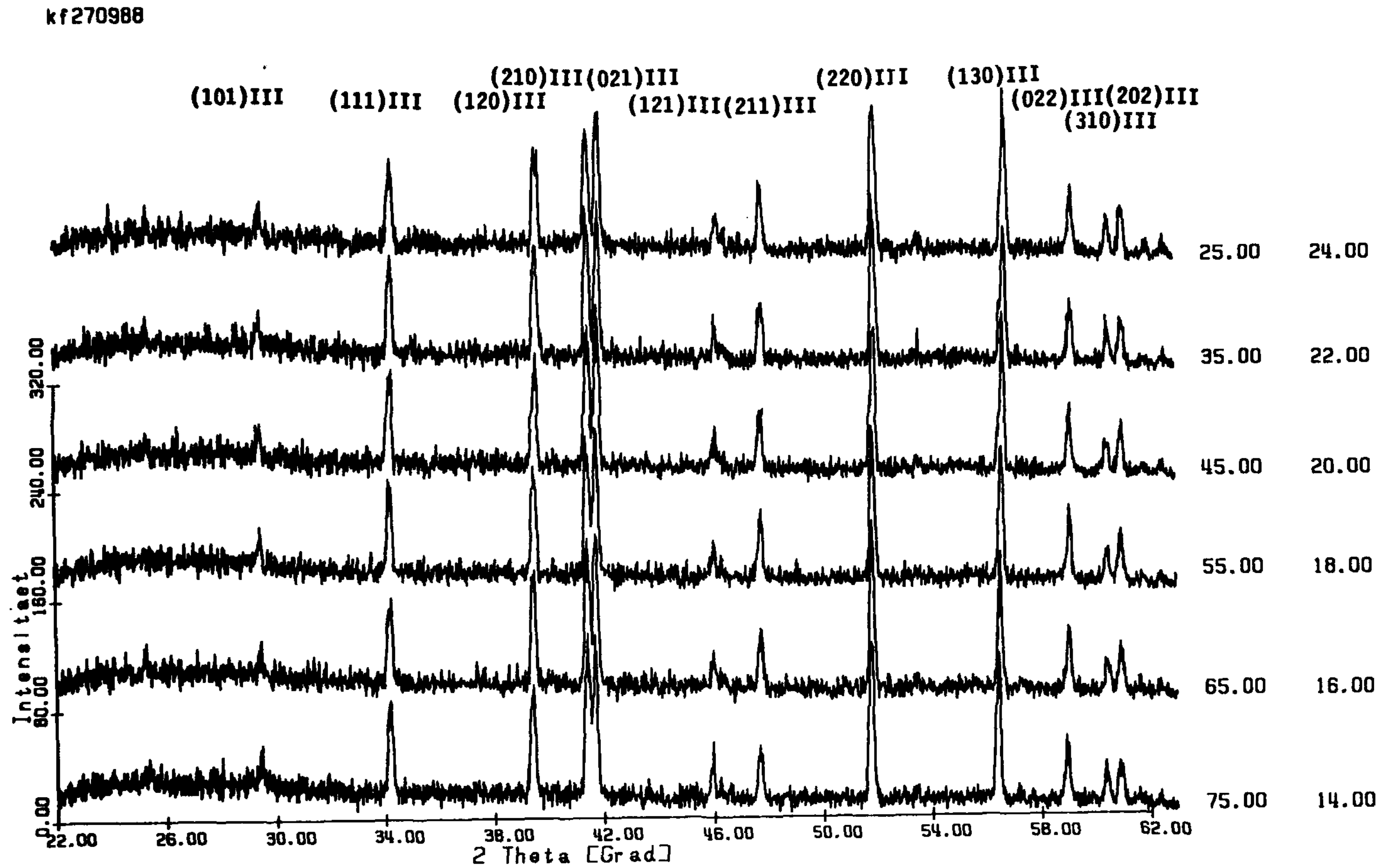
Series
KF 270988
4% KF,dry

Temperature
Program
20/80/−70/80

Diffraction Patterns

kf270988
20/80/-70/80





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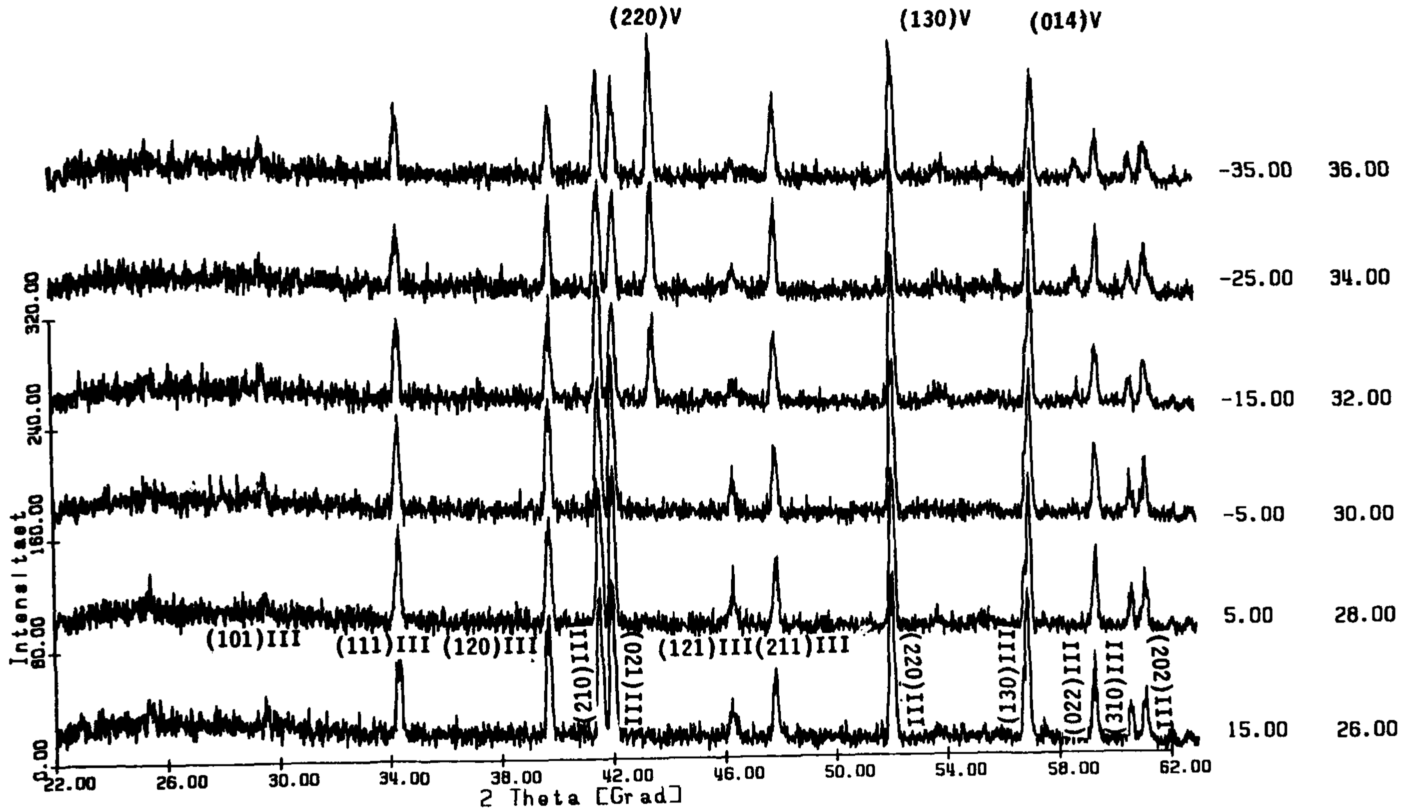

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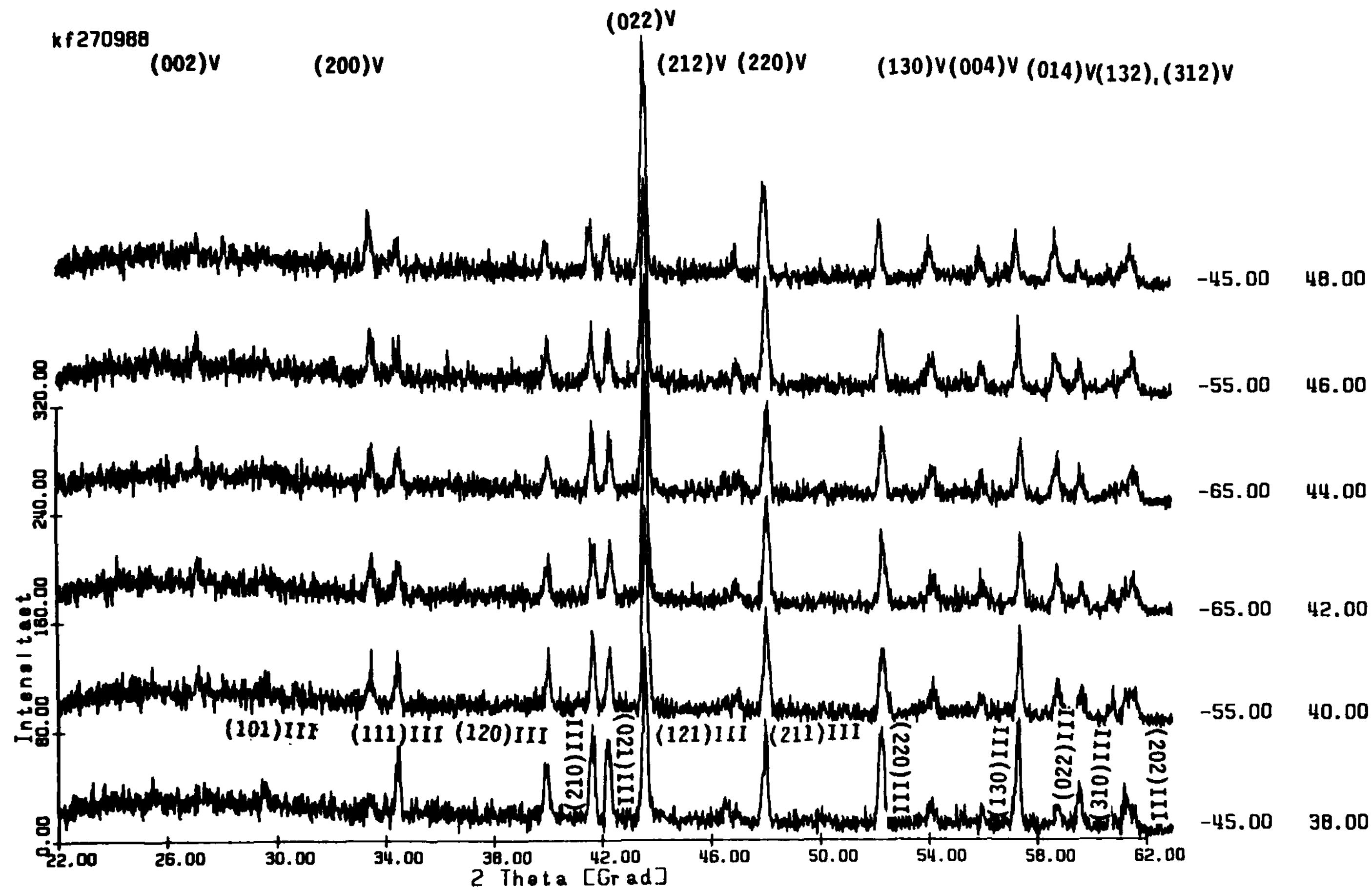
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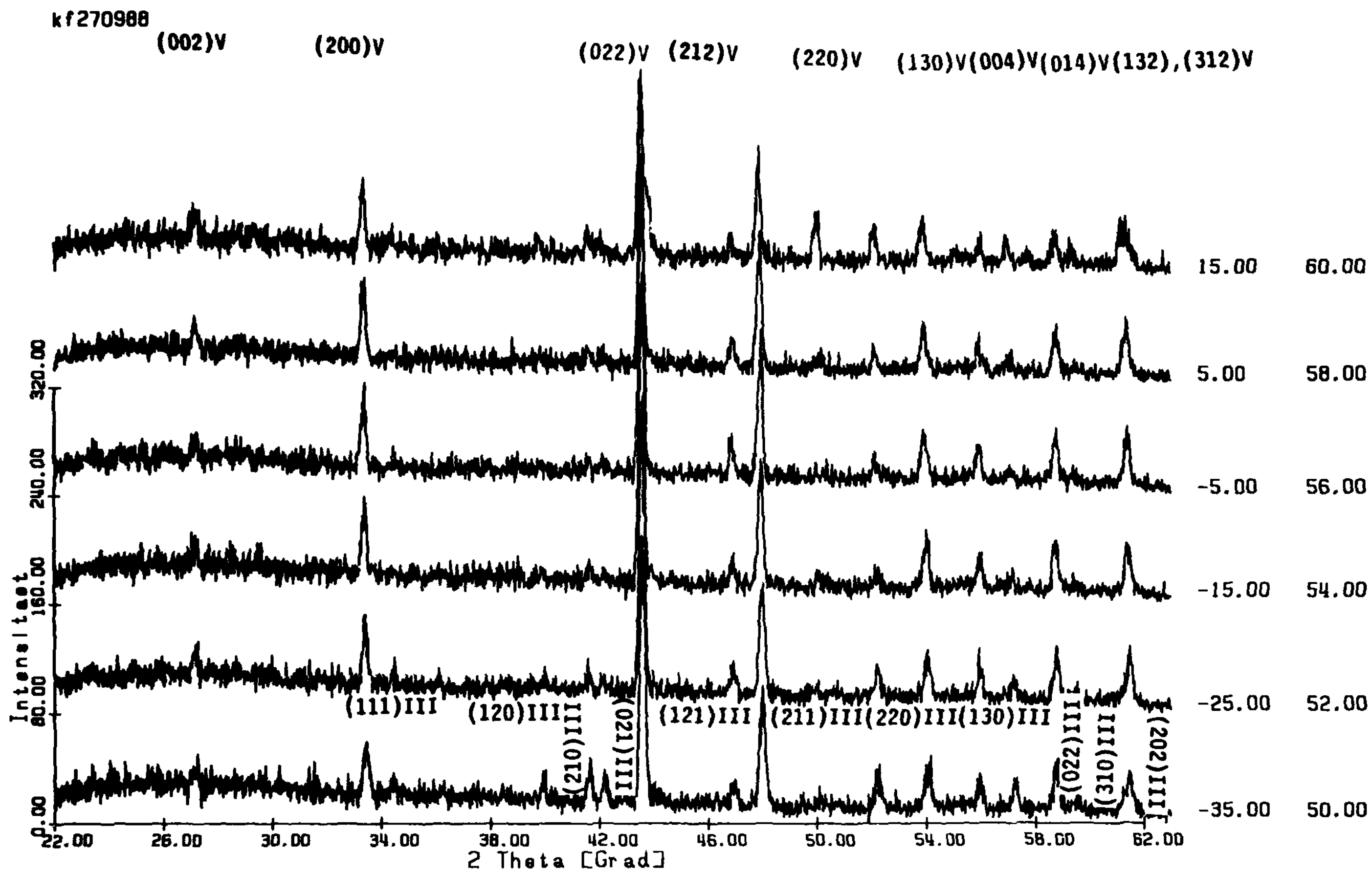
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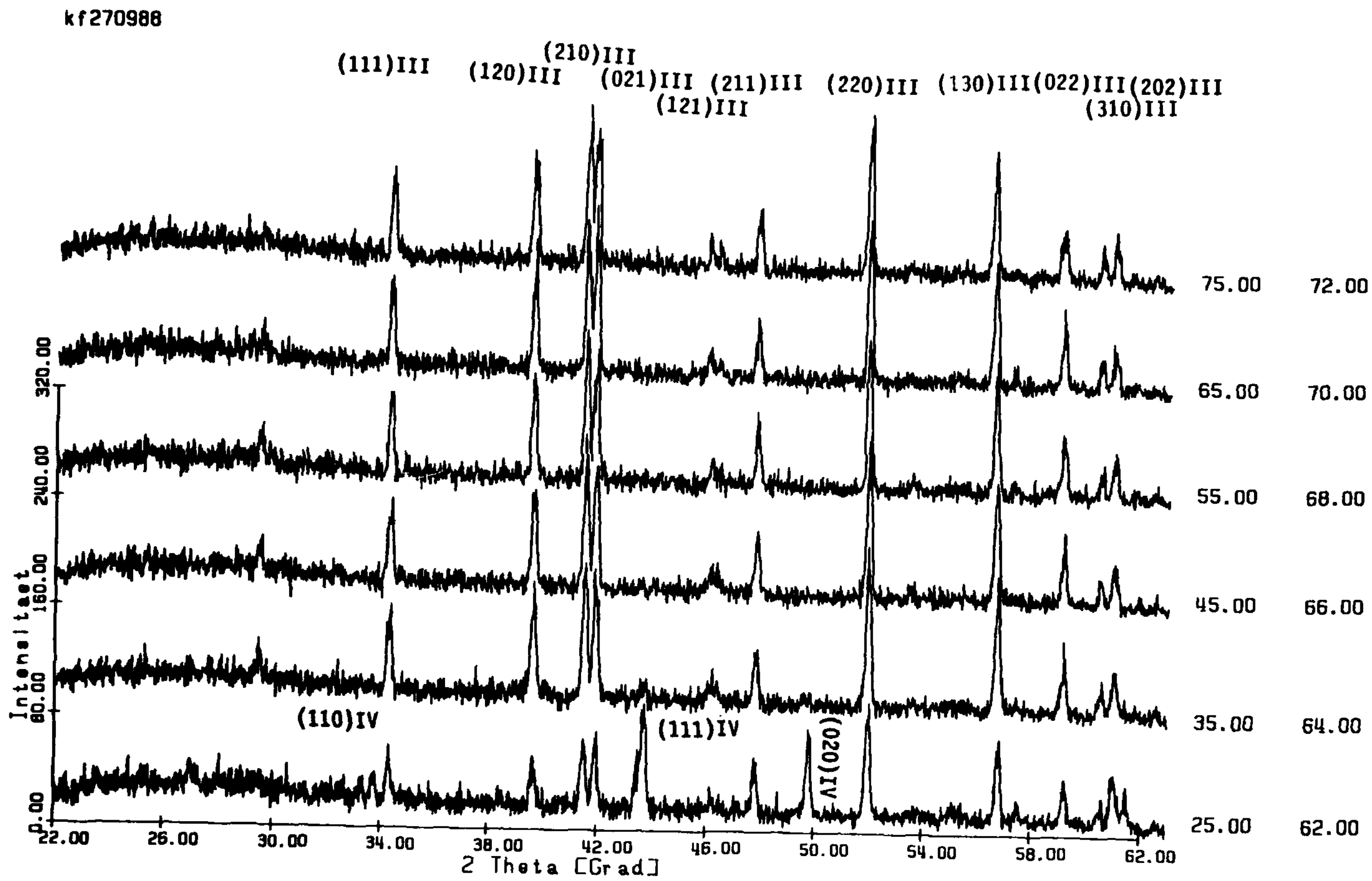

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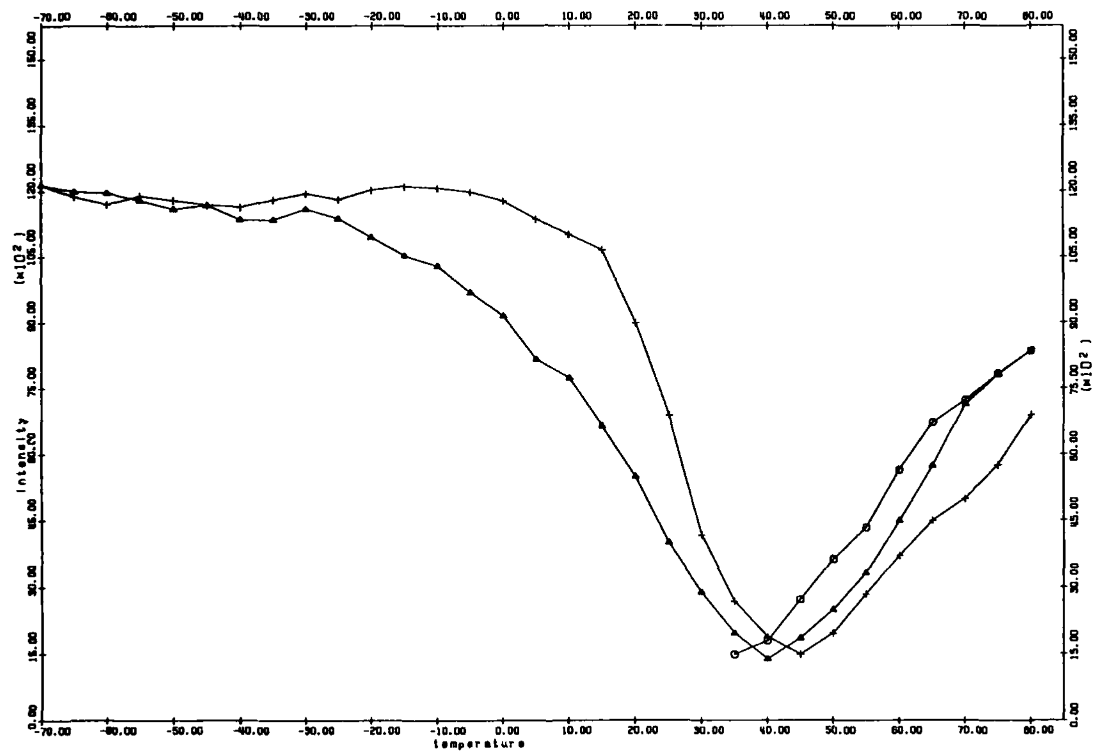
Difference Curve Y(T)

kf270988
20/80/-70/80



Difference Curve dy(T)

kf270988
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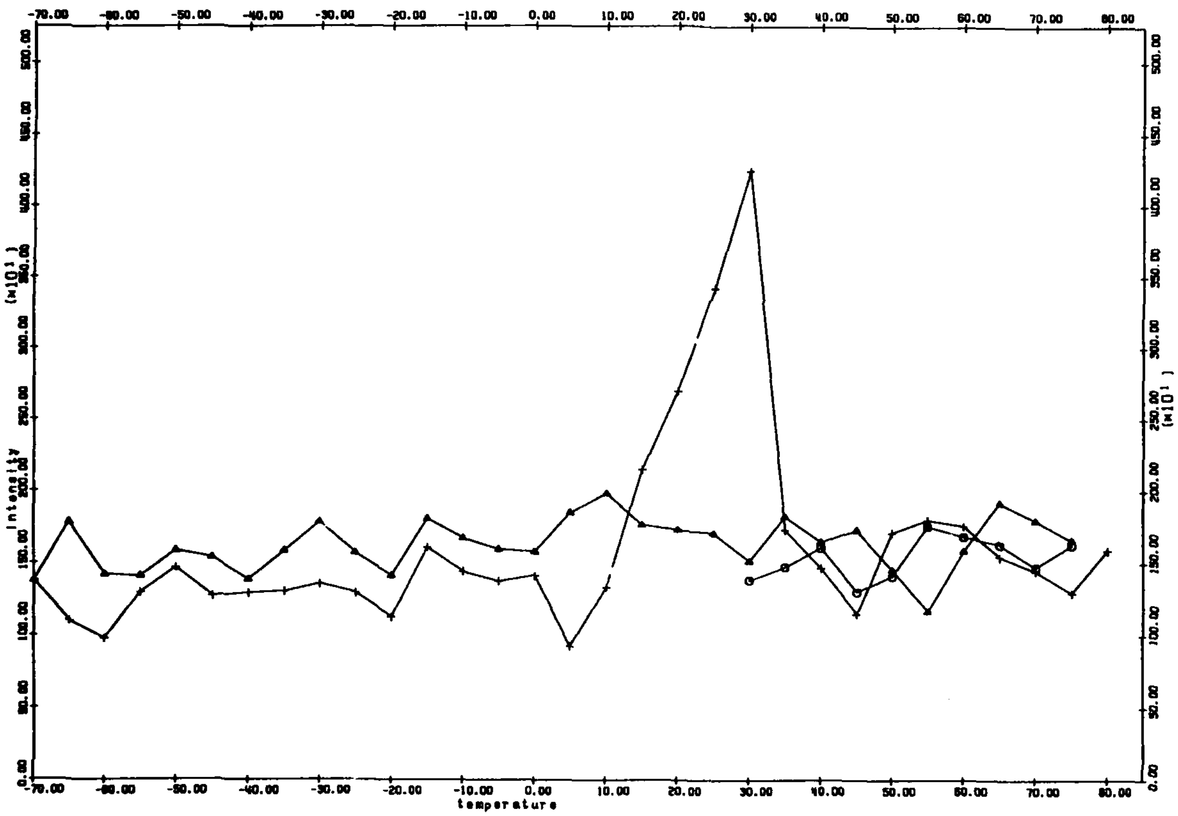
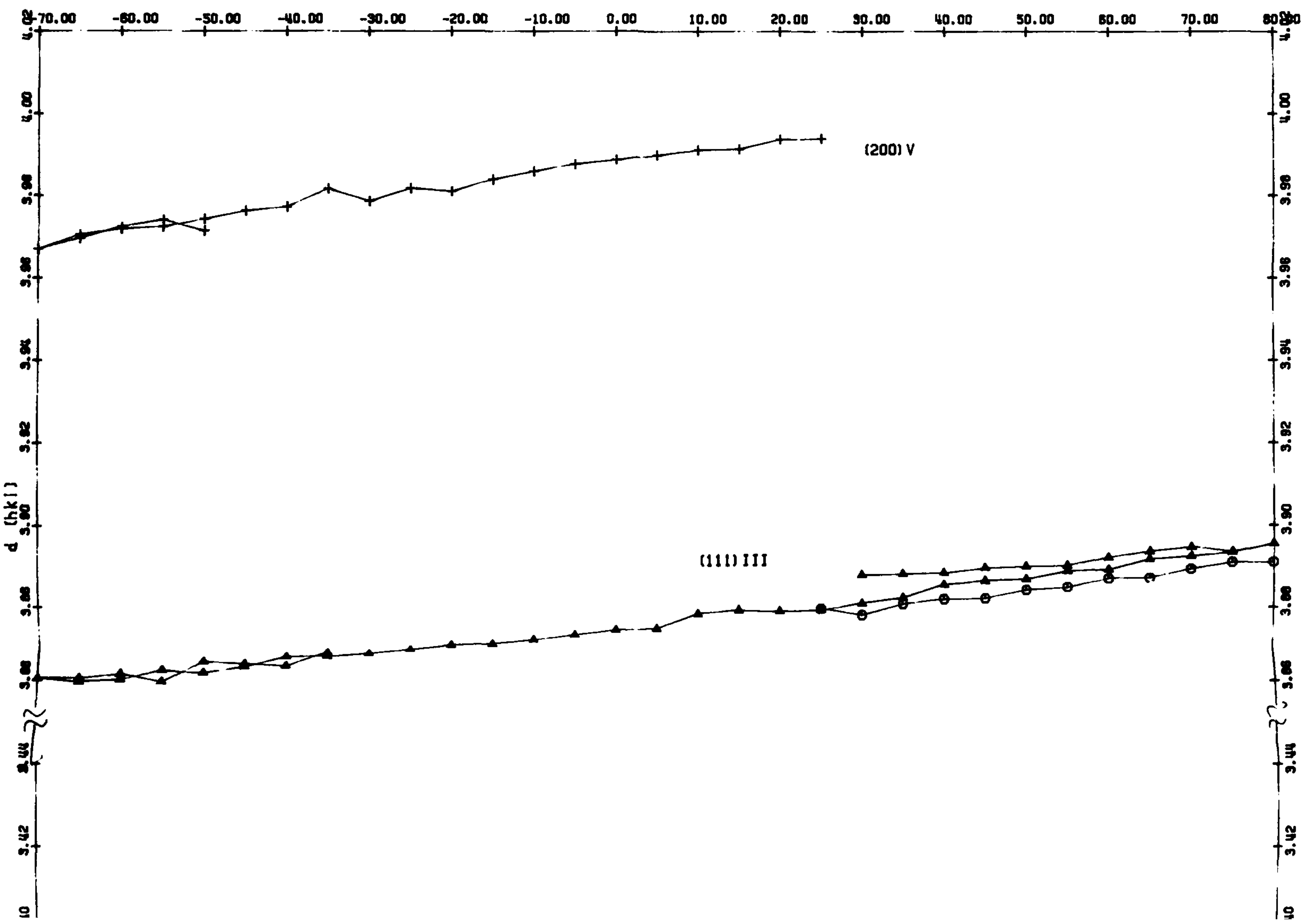
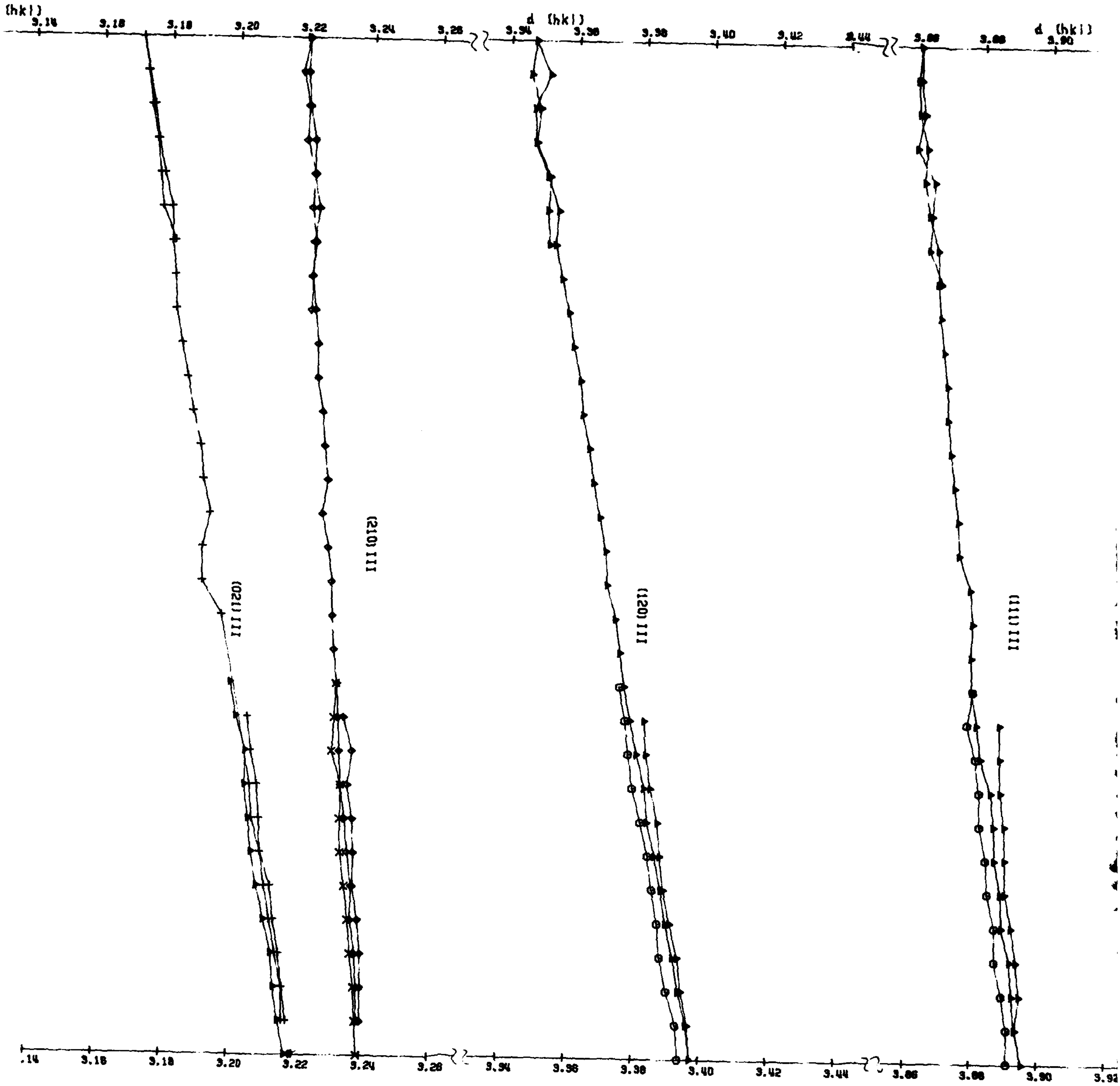


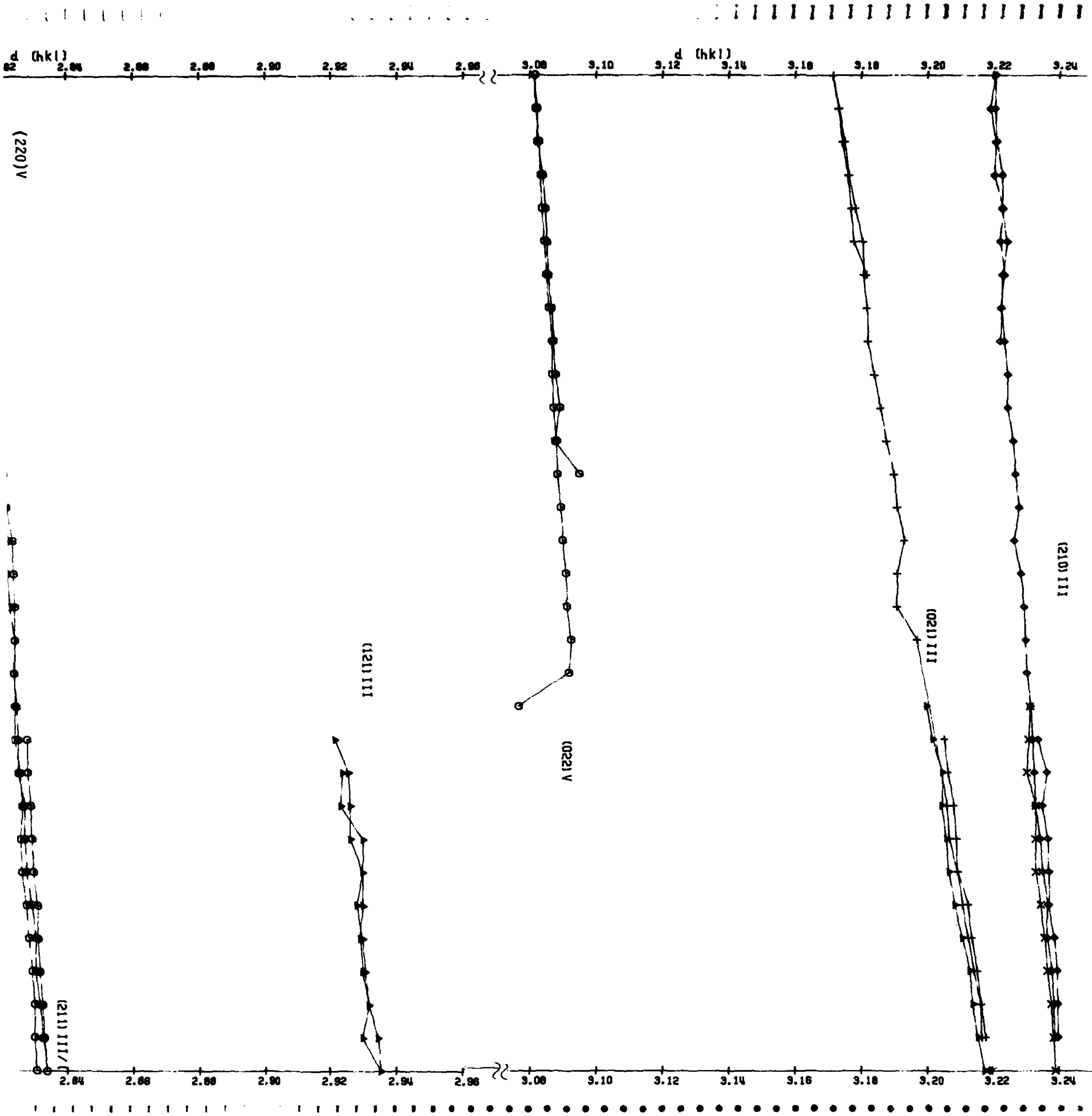
Lattice Plane Distances

kf270988
20/80/-70/20

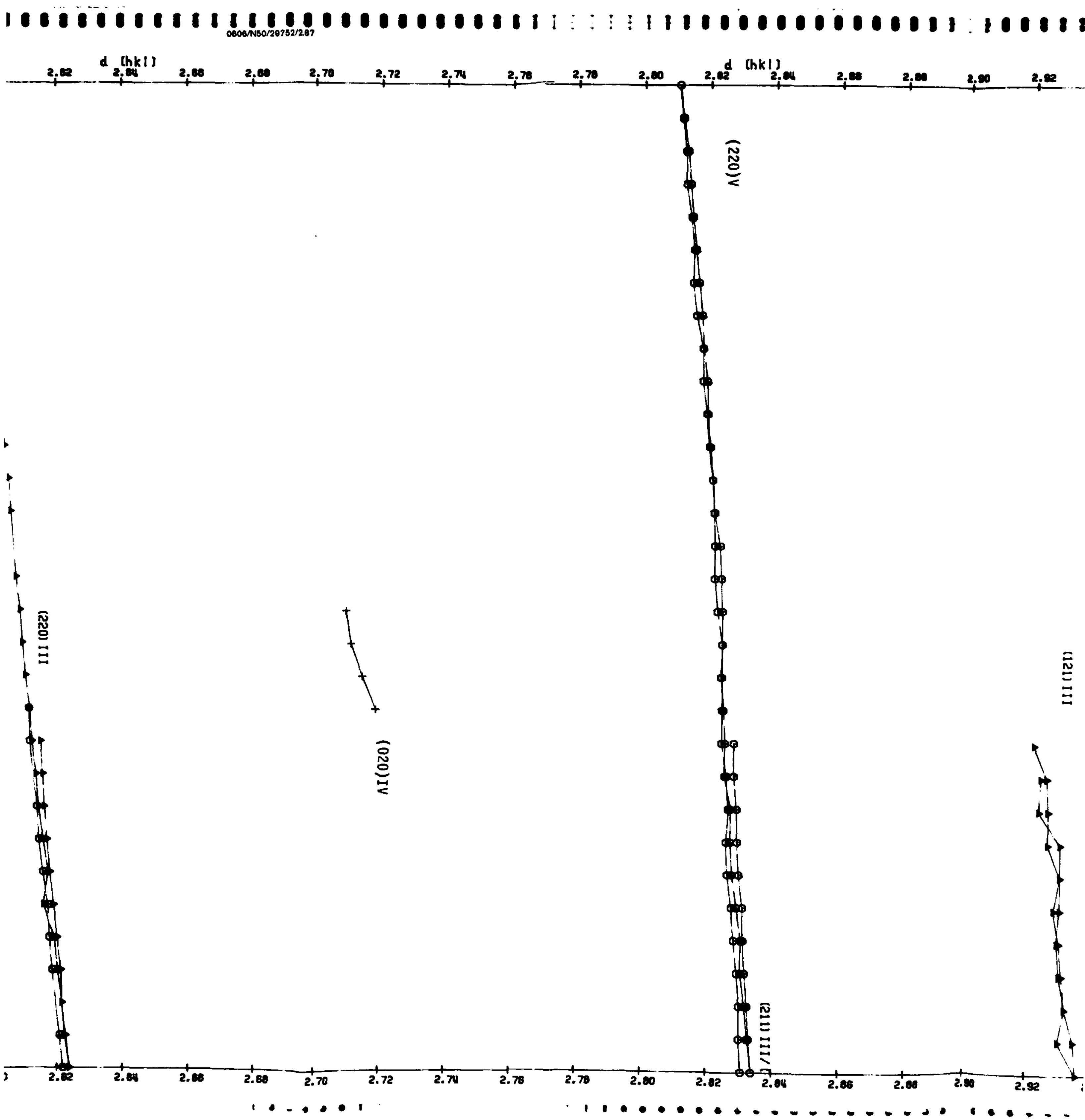


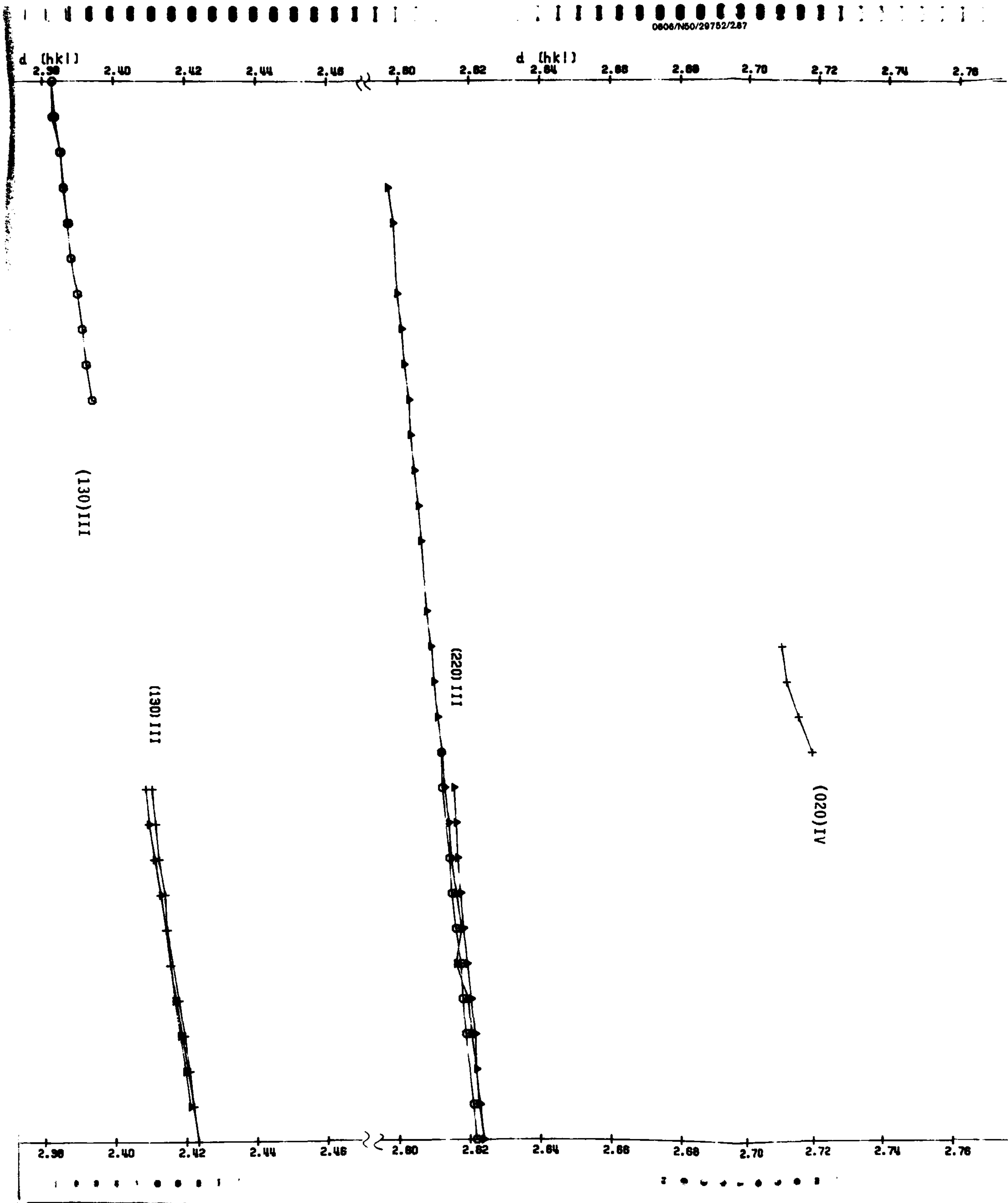
0806/N50/29752/2.87





d (hkl)
2.84
2.86
2.88
2.90
2.92
2.94
2.96
3.08
3.10
3.12
3.14
3.16
3.18
3.20
3.22
3.24
(220)V
(022)V
(121)III
(211)III/(112)
(021)III
(210)III

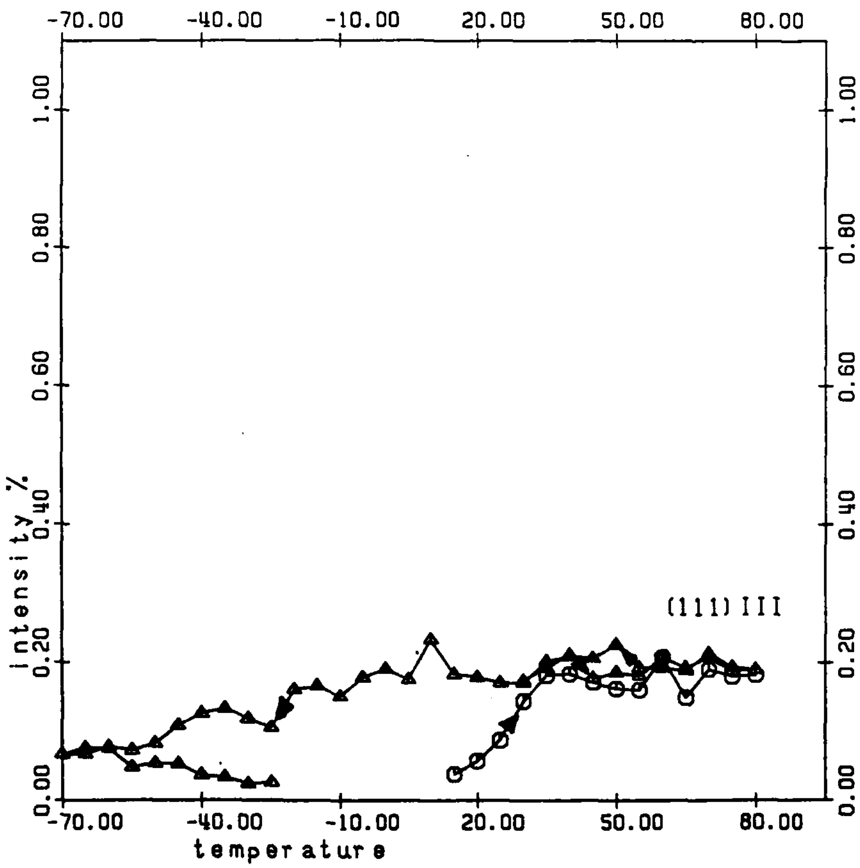

0606/N50/29752/2.67
d (hkl)
d (hkl)
2.82
2.84
2.86
2.68
2.70
2.72
2.74
2.76
2.78
2.80
2.82
2.84
2.86
2.88
2.90
2.92
(220)V
(220)III
(020)IV
(121)III
(211)III/


0606/N50/29752/2.87
d (hkl)
2.38
2.40
2.42
2.44
2.46
2.80
2.82
d (hkl)
2.64
2.66
2.68
2.70
2.72
2.74
2.76
(130)III
(220)III
(130)III
(020)IV
2.38
2.40
2.42
2.44
2.46
2.80
2.82
2.64
2.66
2.68
2.70
2.72
2.74
2.76

Intensities

kf270988

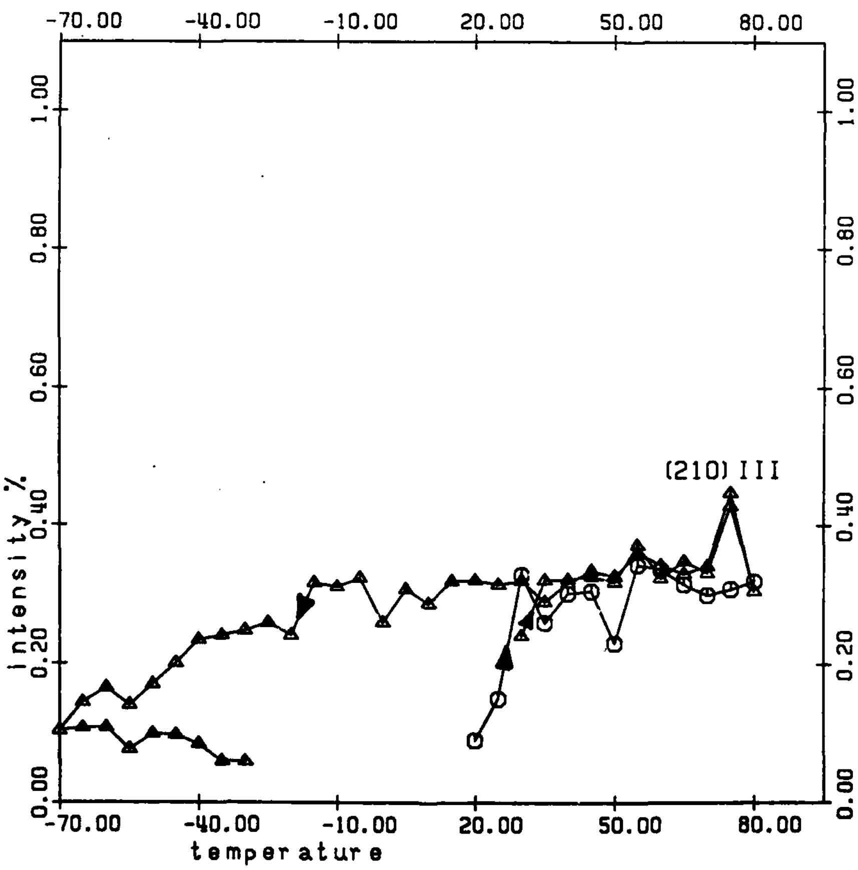
4% KF dry 20/80/-70/80



Intensities

kf270988

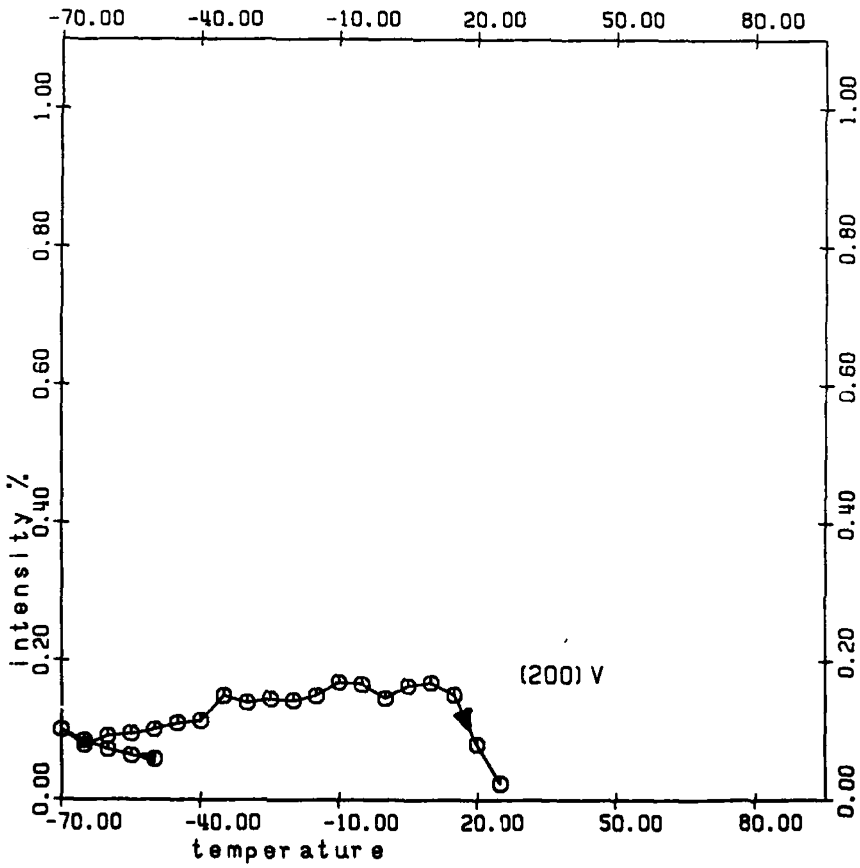
4% KF dry 20/80/-70/80



Intensities

kf270988

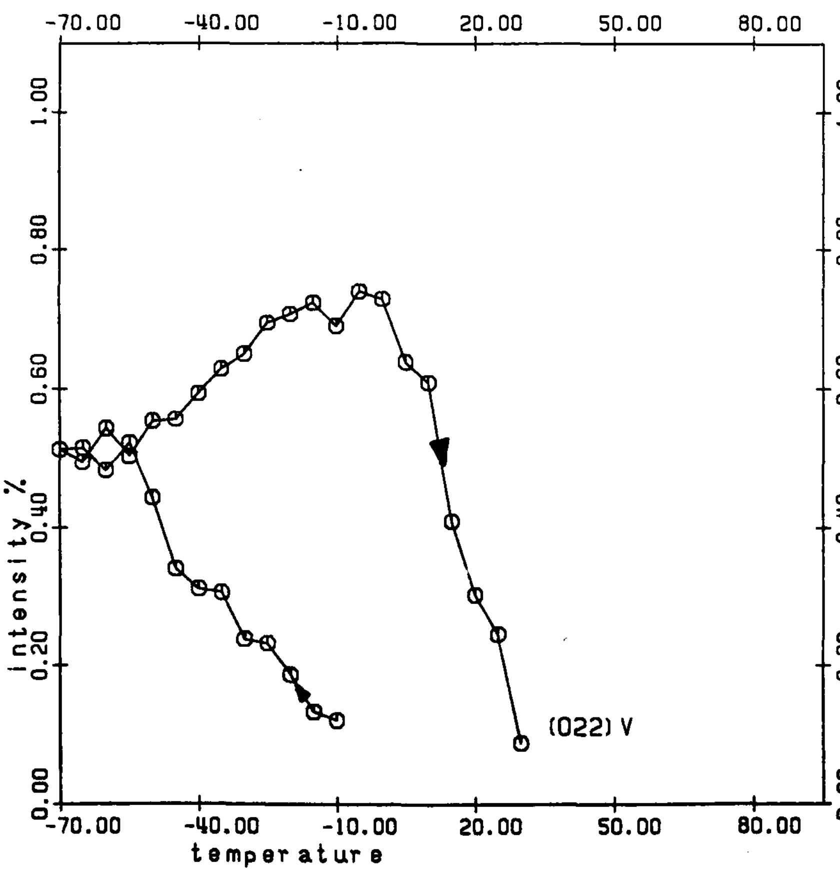
4% KF dry 20/80/-70/80



Intensities

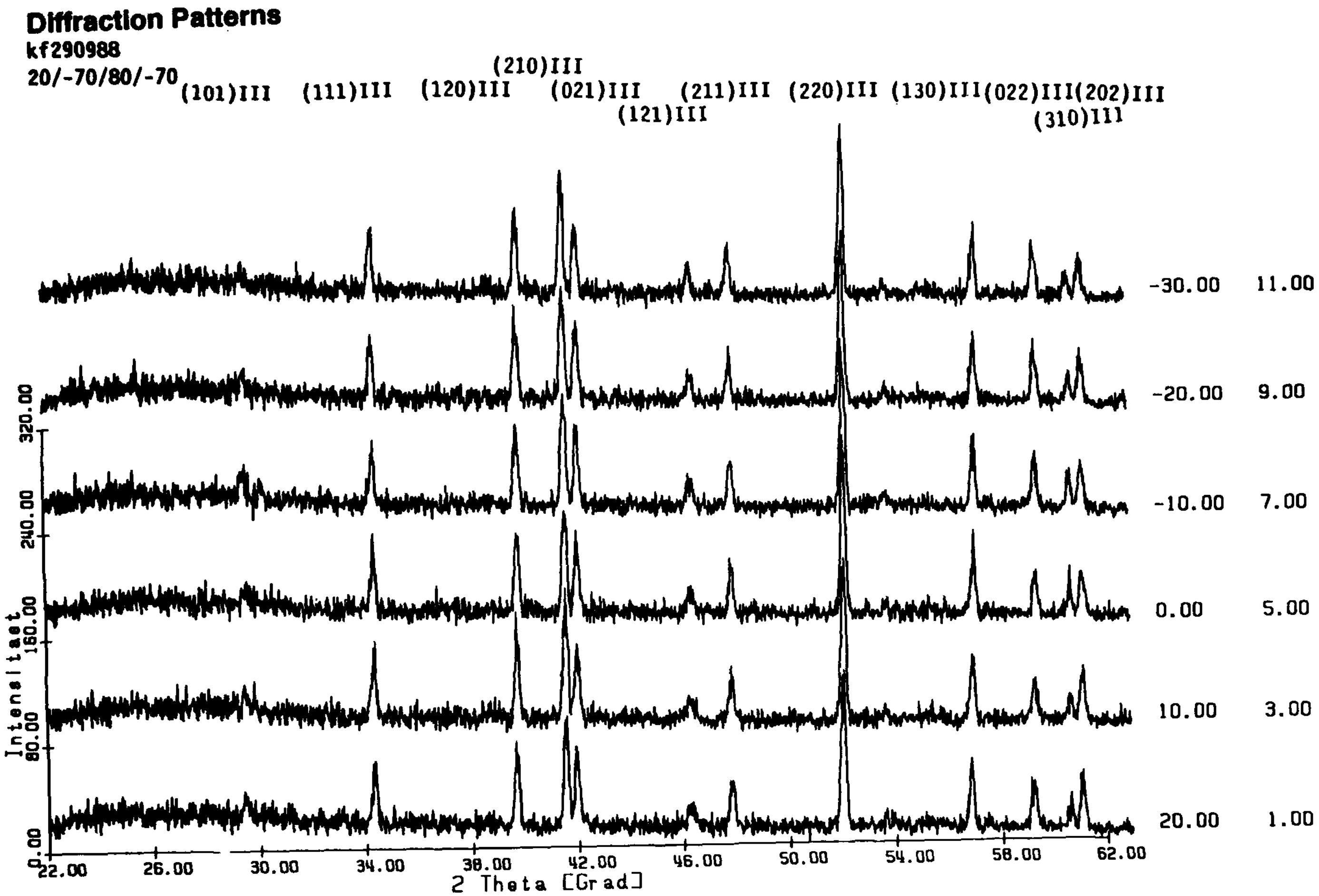
kf270988

4% KF dry 20/80/-70/80



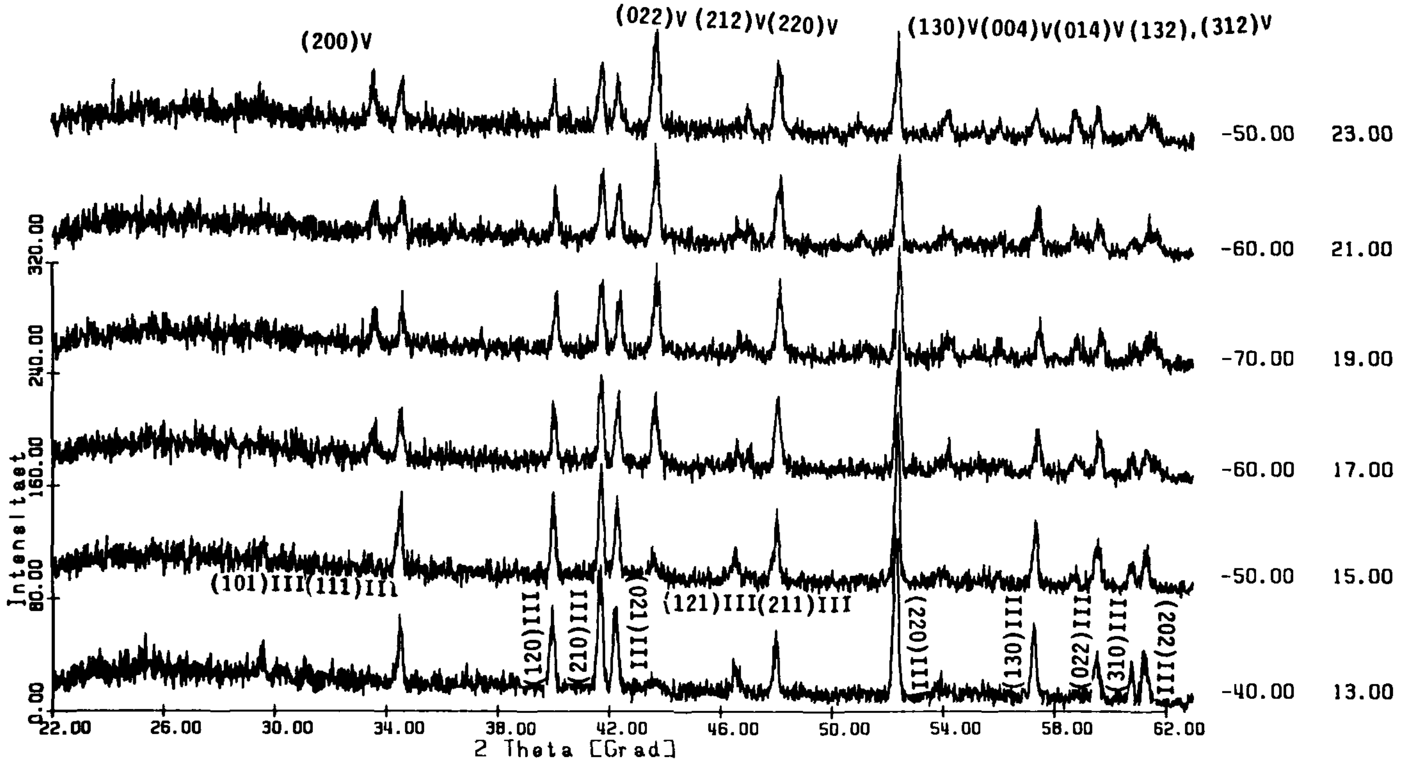
Series
KF 290988
4% KF,dry

Temperature
Program
20/–70/80/–70

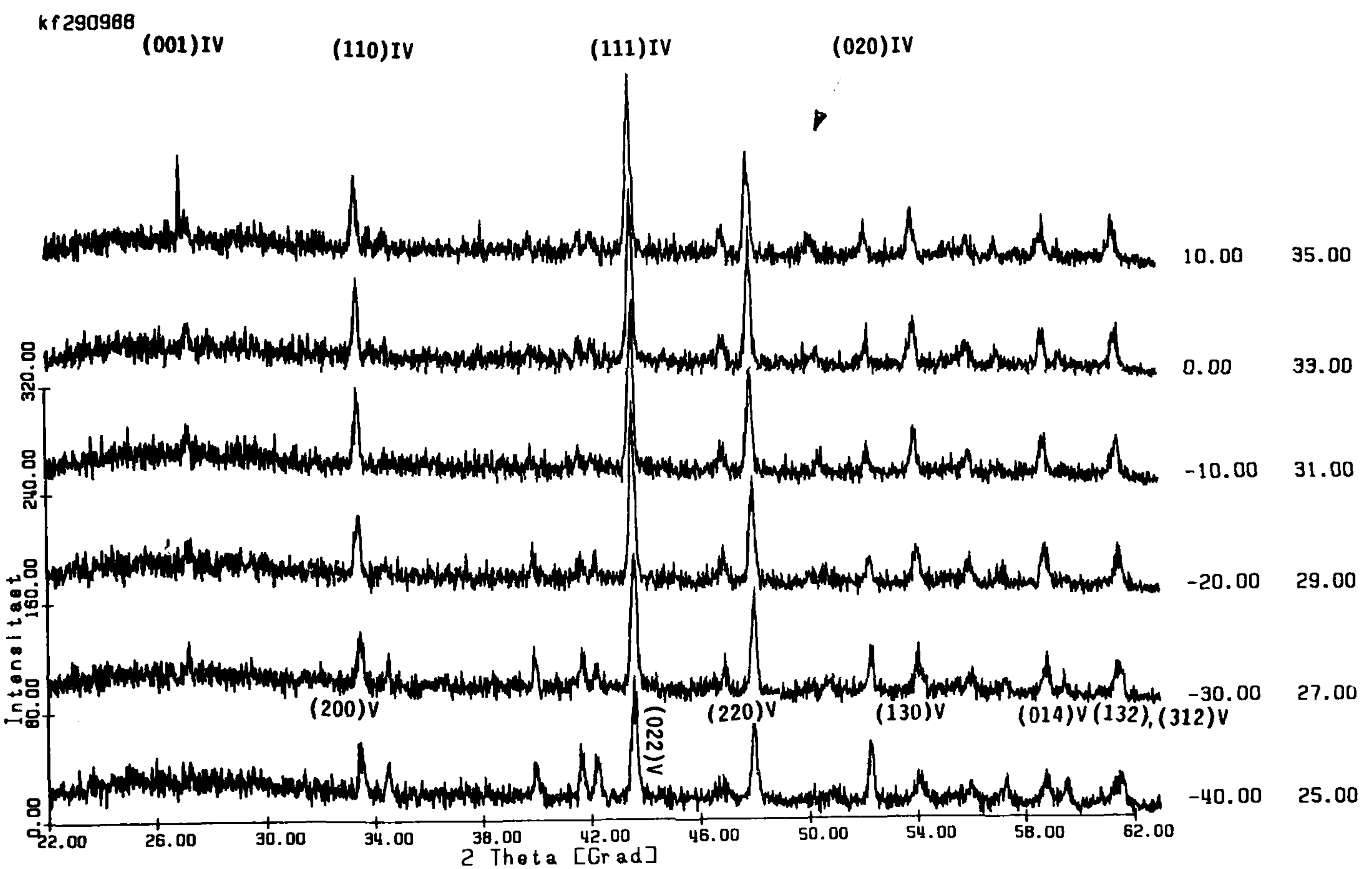


Diffraction Patterns
kf290988
20/-70/80/-70
(101)III
(111)III
(120)III
(210)III
(021)III
(121)III
(211)III
(220)III
(130)III
(022)III
(202)III
(310)III
-30.00 11.00
-20.00 9.00
-10.00 7.00
0.00 5.00
10.00 3.00
20.00 1.00
Intensitaet
0.00
80.00
160.00
240.00
320.00
22.00
26.00
30.00
34.00
38.00
42.00
46.00
50.00
54.00
58.00
62.00
2 Theta [Grad]

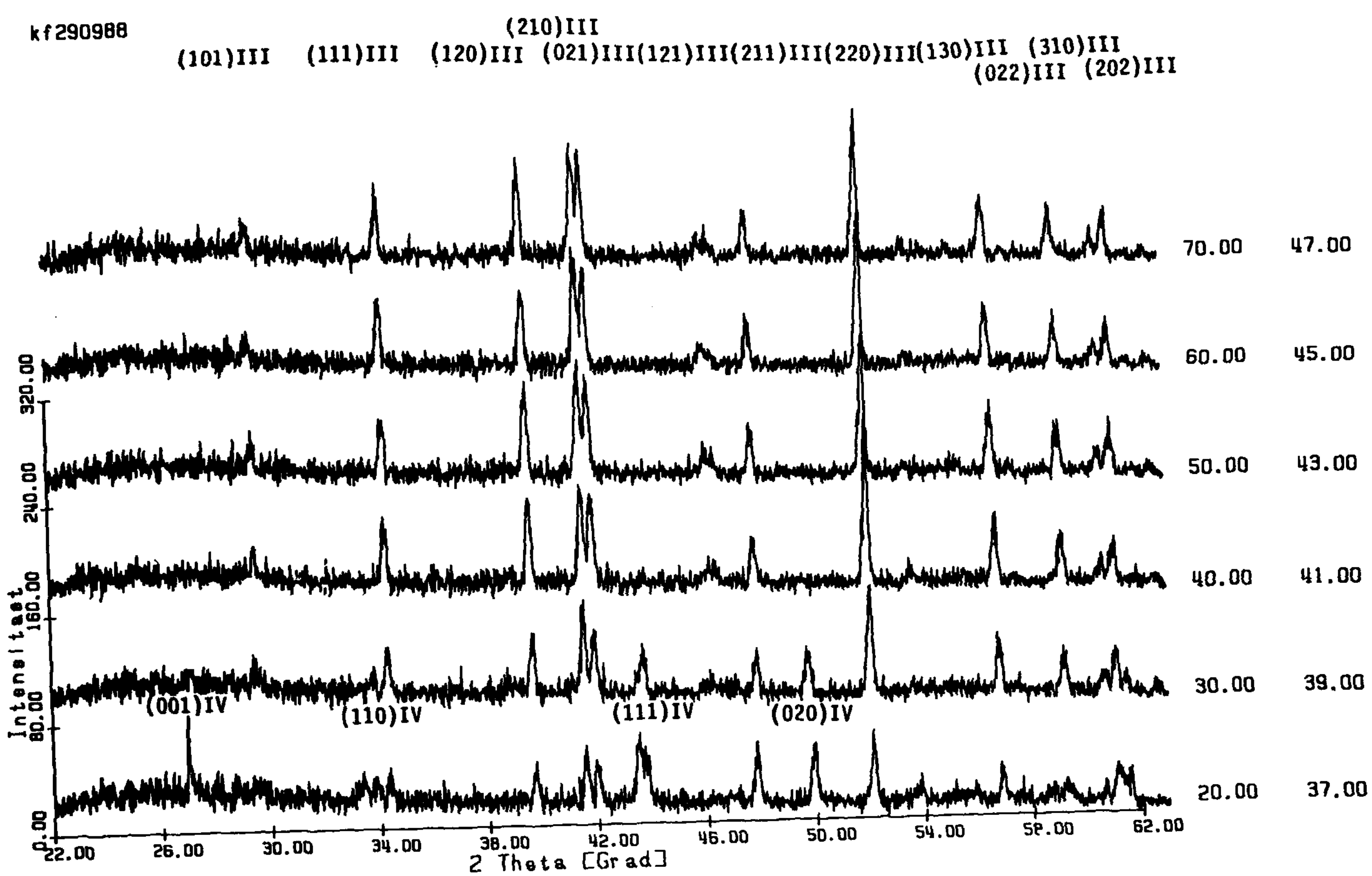
kf290988



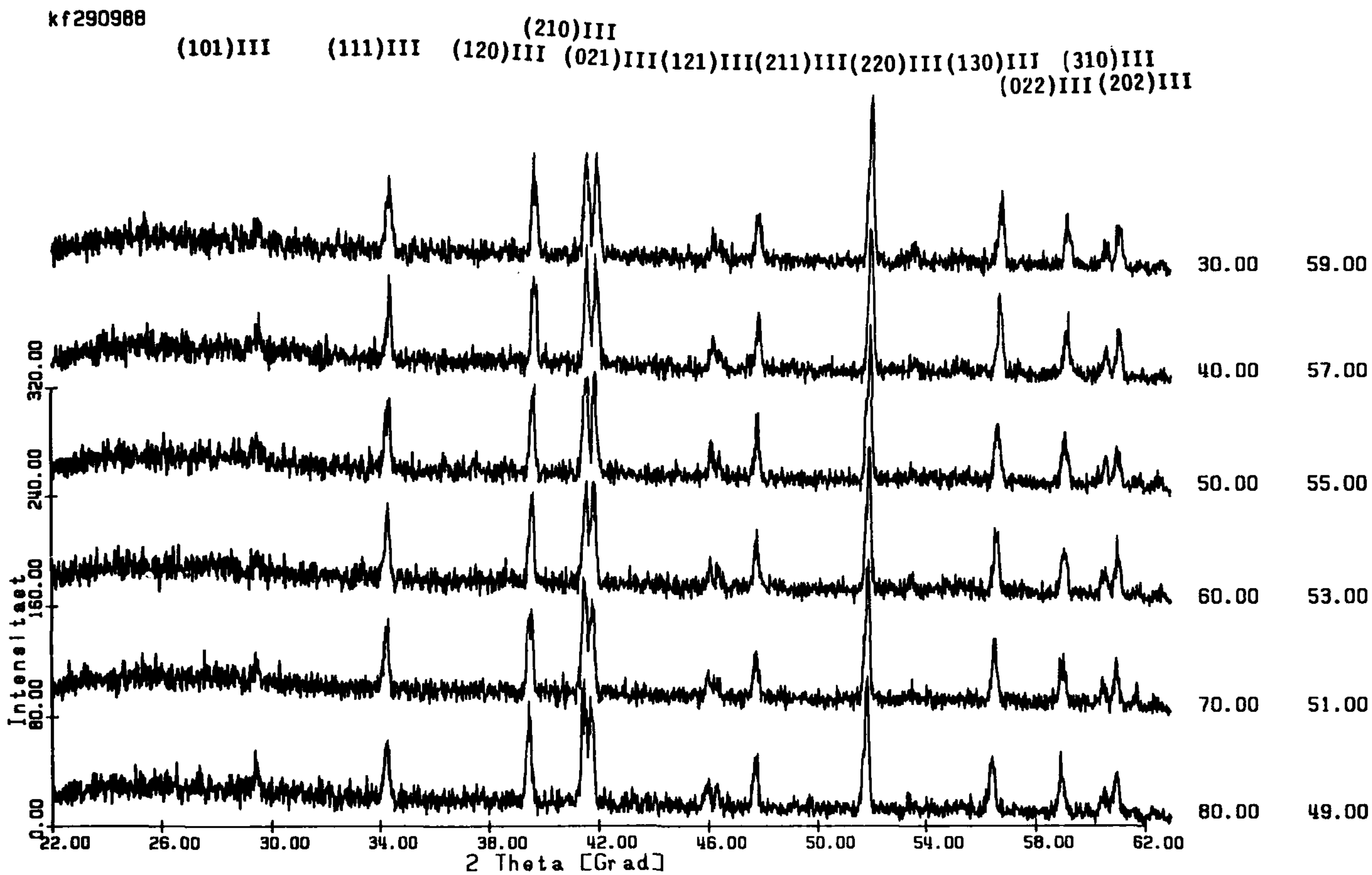
(200)V
(022)V (212)V(220)V
(130)V(004)V(014)V(132),(312)V
-50.00 23.00
-60.00 21.00
-70.00 19.00
-60.00 17.00
-50.00 15.00
-40.00 13.00
(101)III(111)III
(120)III
(210)III
(021)III
(121)III(211)III
(220)III
(130)III
(022)III
(310)III
(202)III
Intensitaet
0.00
80.00
160.00
240.00
320.00
22.00
26.00
30.00
34.00
38.00
42.00
46.00
50.00
54.00
58.00
62.00
2 Theta [Grad]

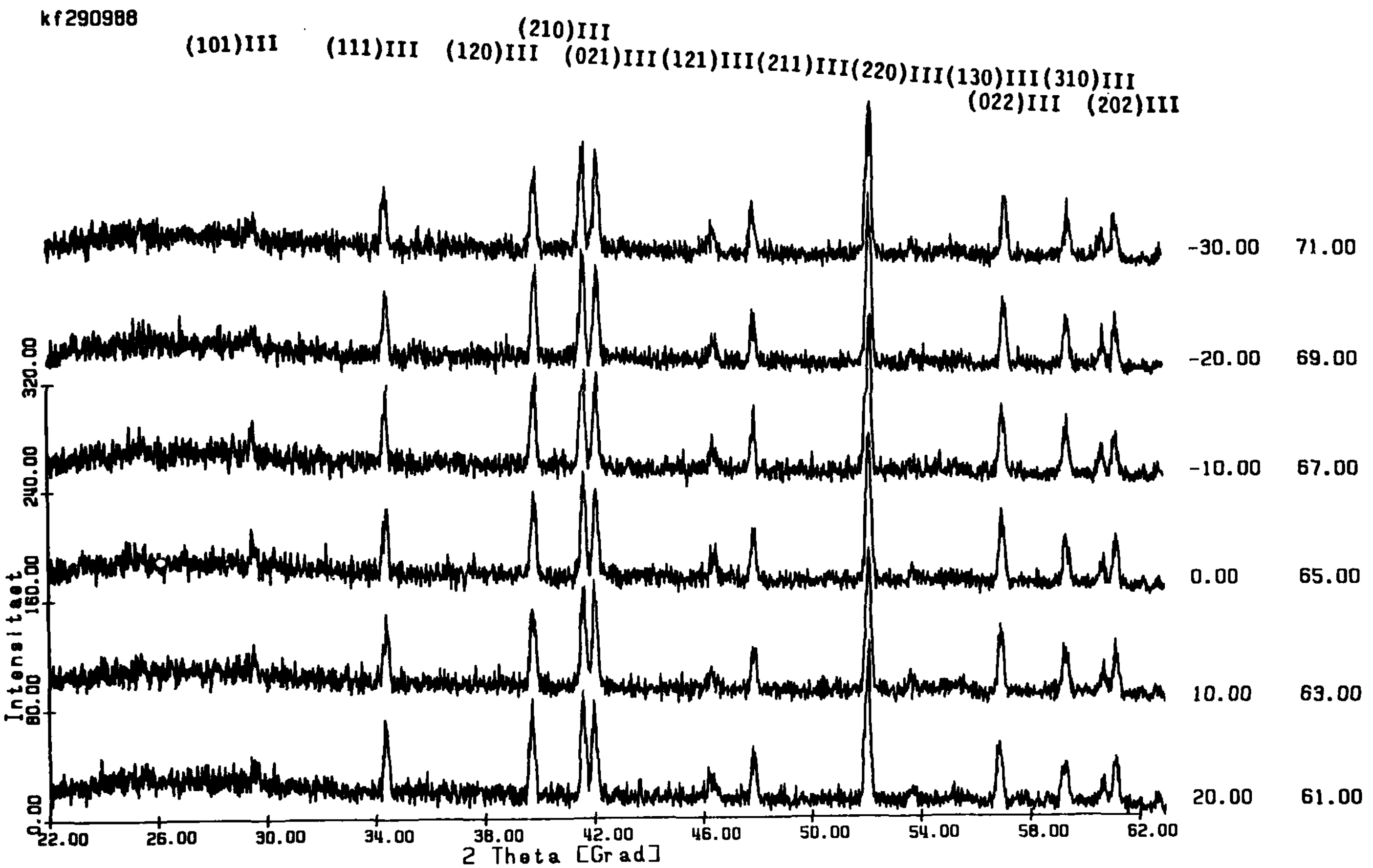


kf290988
(001)IV
(110)IV
(111)IV
(020)IV
(200)V
(022)V
(220)V
(130)V
(014)V (132), (312)V
10.00 35.00
0.00 33.00
-10.00 31.00
-20.00 29.00
-30.00 27.00
-40.00 25.00
Intensitaet
0.00
80.00
160.00
240.00
320.00
22.00
26.00
30.00
34.00
38.00
42.00
46.00
50.00
54.00
58.00
62.00
2 Theta [Grad]

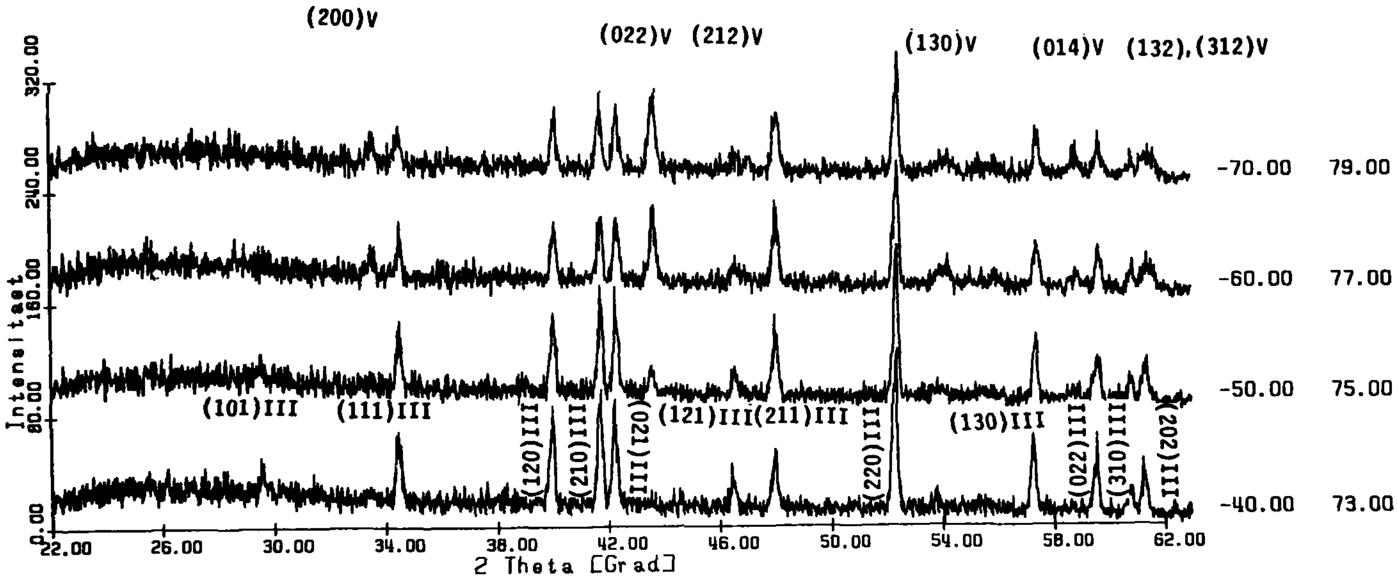


kf290988
(210)III
(101)III
(111)III
(120)III
(021)III(121)III(211)III(220)III(130)III
(310)III
(022)III
(202)III
(001)IV
(110)IV
(111)IV
(020)IV
70.00 47.00
60.00 45.00
50.00 43.00
40.00 41.00
30.00 39.00
20.00 37.00
Intensitaet
0.00
80.00
160.00
240.00
320.00
22.00
26.00
30.00
34.00
38.00
42.00
46.00
50.00
54.00
58.00
62.00
2 Theta [Grad]


kf290988
(101)III
(111)III
(120)III
(210)III
(021)III
(121)III
(211)III
(220)III
(130)III
(310)III
(022)III
(202)III
30.00 59.00
40.00 57.00
50.00 55.00
60.00 53.00
70.00 51.00
80.00 49.00
Intensitaet
0.00
80.00
160.00
240.00
320.00
22.00
26.00
30.00
34.00
38.00
42.00
46.00
50.00
54.00
58.00
62.00
2 Theta [Grad]


kf290988
(101)III
(111)III
(120)III
(210)III
(021)III
(121)III
(211)III
(220)III
(130)III
(310)III
(022)III
(202)III
-30.00 71.00
-20.00 69.00
-10.00 67.00
0.00 65.00
10.00 63.00
20.00 61.00
Intensitaet
0.00
80.00
160.00
240.00
320.00
22.00
26.00
30.00
34.00
38.00
42.00
46.00
50.00
54.00
58.00
62.00
2 Theta [Grad]

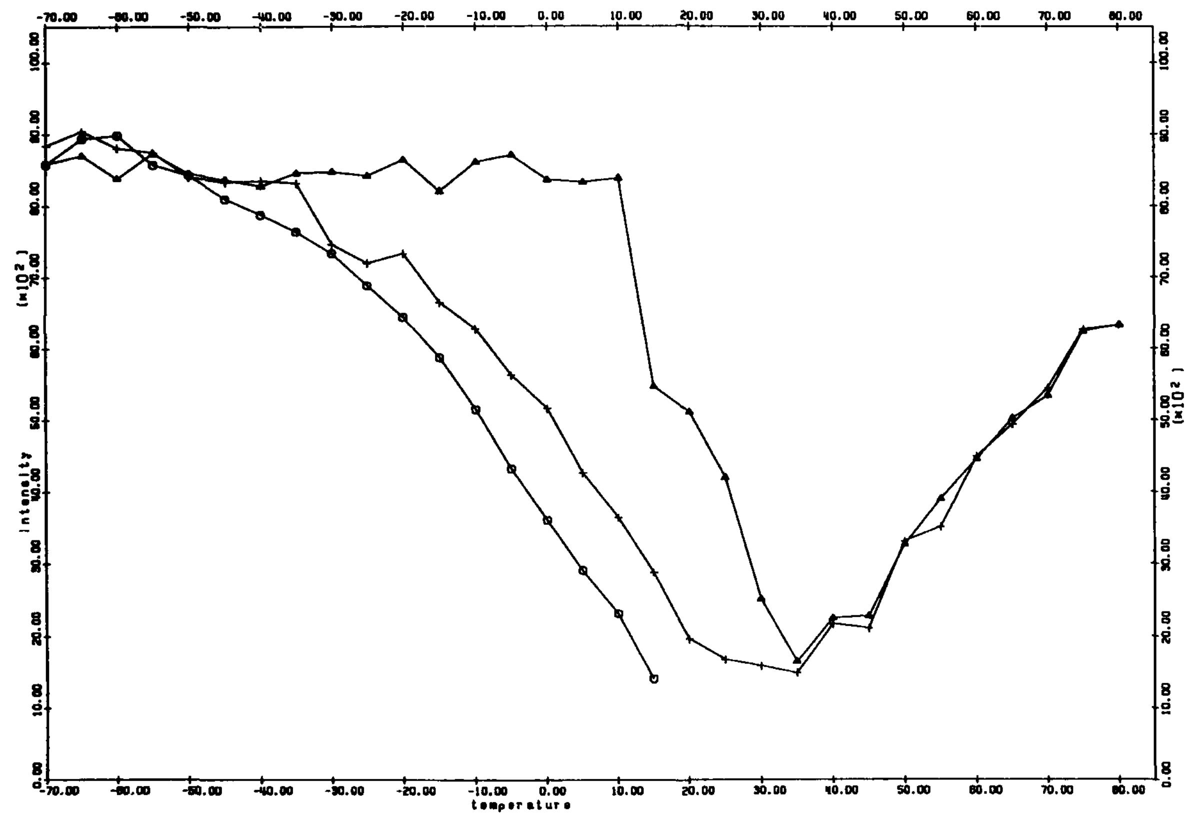
kf290988



(200)V
(022)V (212)V
(130)V
(014)V (132),(312)V
(101)III (111)III
(120)III (210)III (021)III (121)III (211)III (220)III
(130)III (022)III (310)III (202)III
-70.00 79.00
-60.00 77.00
-50.00 75.00
-40.00 73.00
Intensitaet
0.00
80.00
160.00
240.00
320.00
22.00 26.00 30.00 34.00 38.00 42.00 46.00 50.00 54.00 58.00 62.00
2 Theta [Grad]

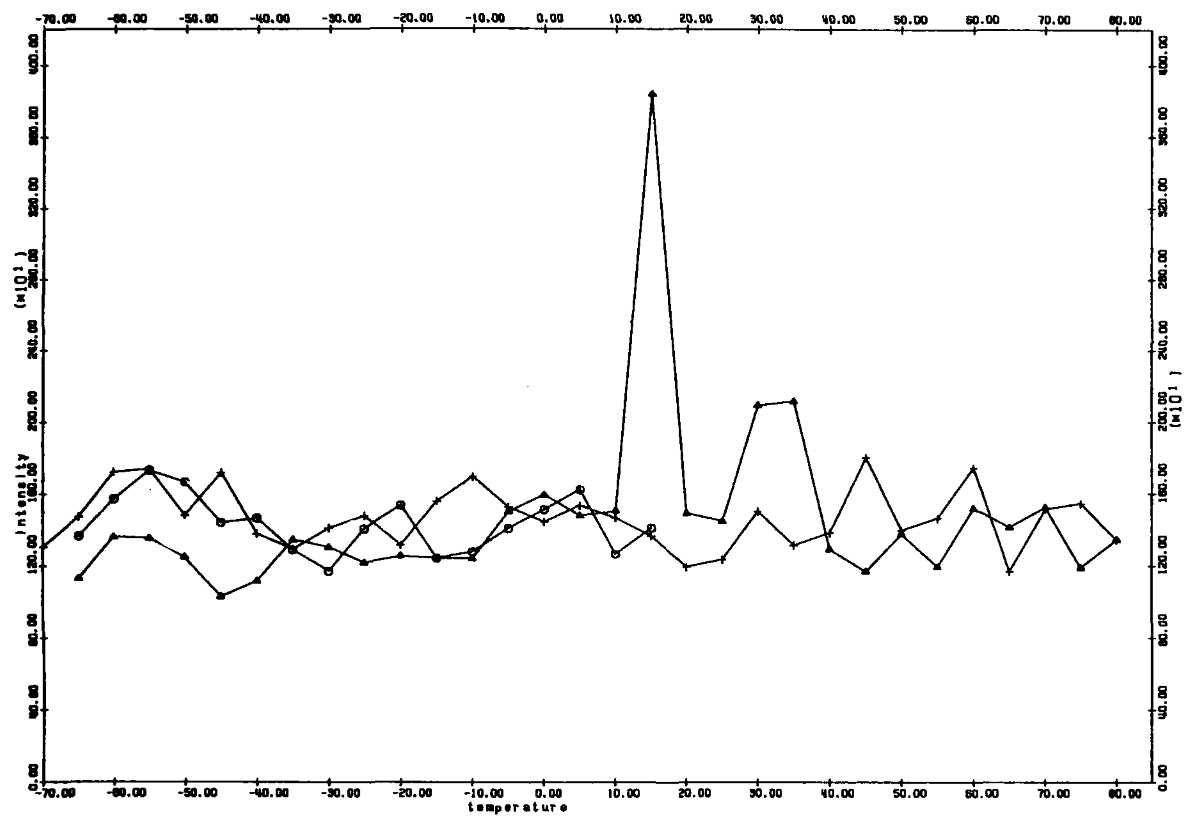
Difference Curve Y(T)

kf290988
20/-70/80/-70



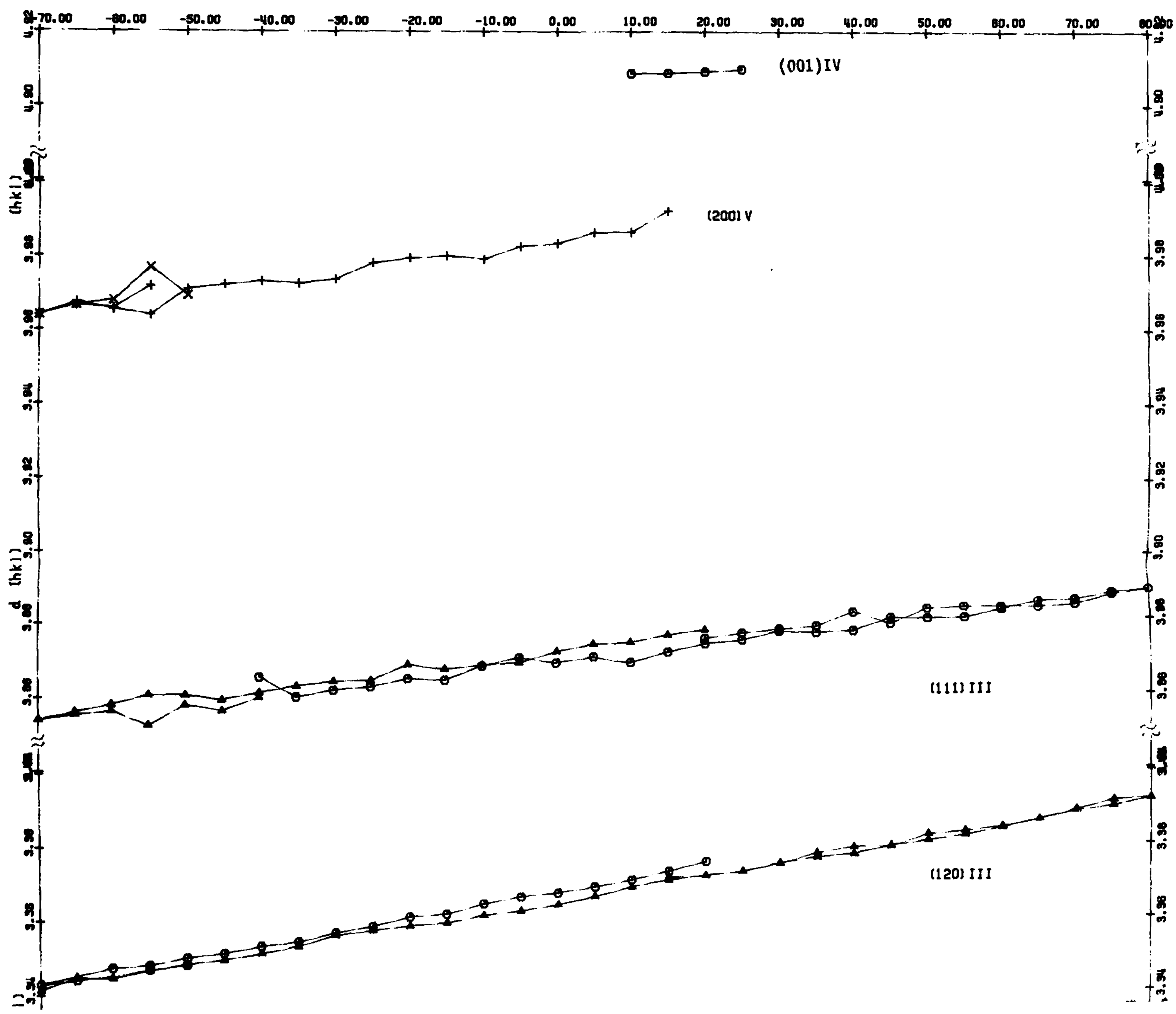
Difference Curve dy(T)

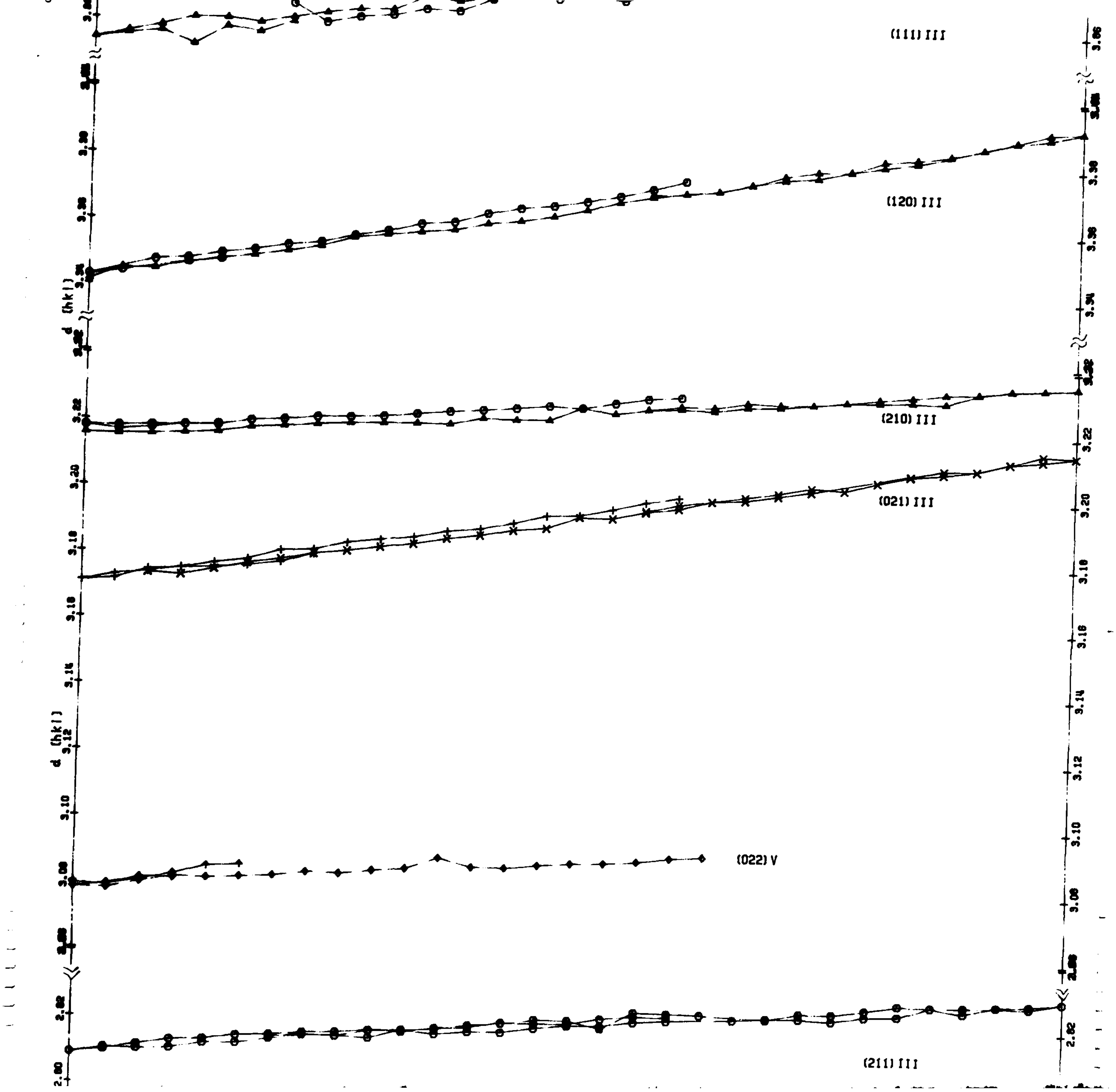
kf290988
20/-70/80/-70

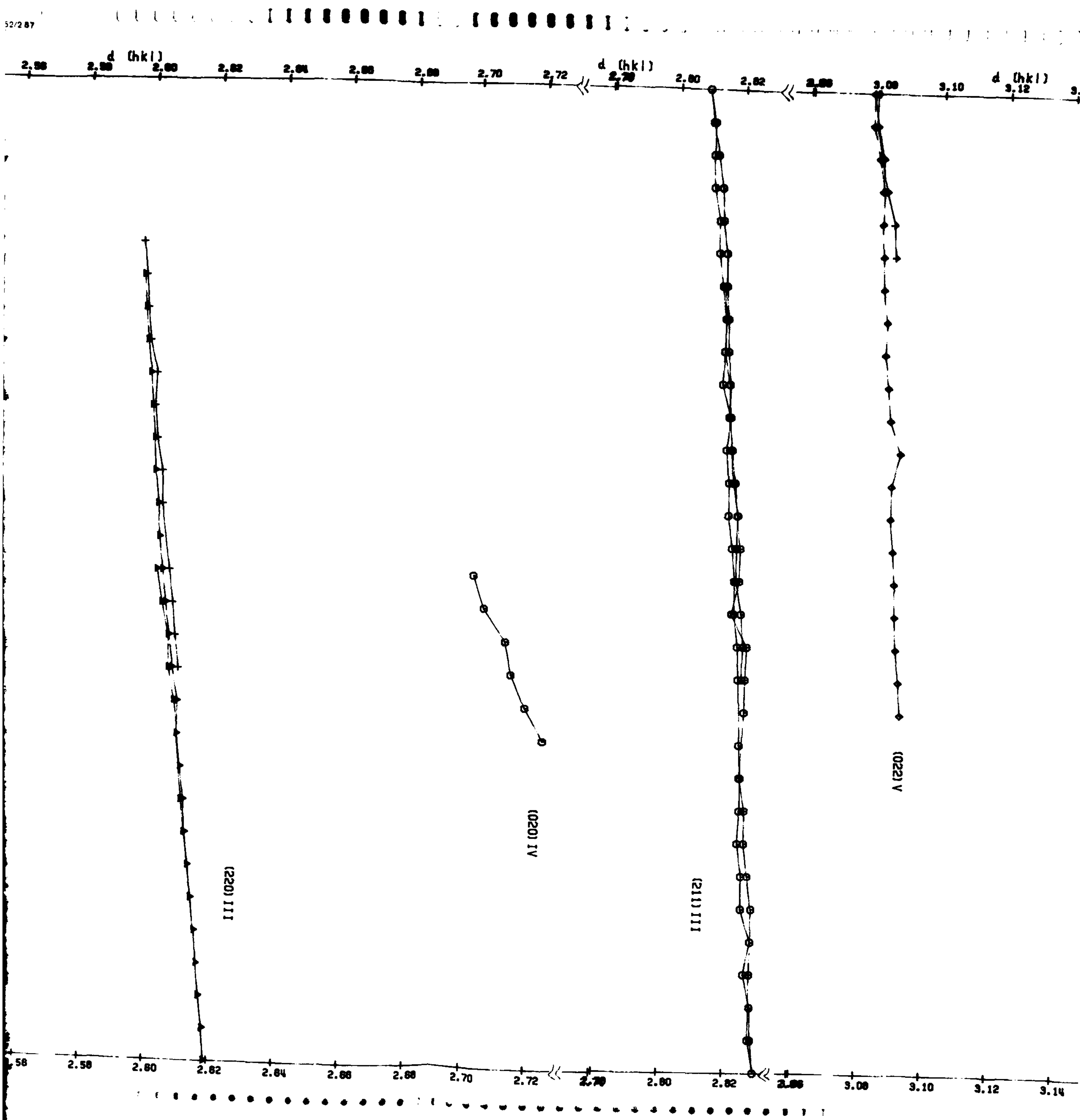


Lattice Plane Distances

kf290988
20/-70/80/20

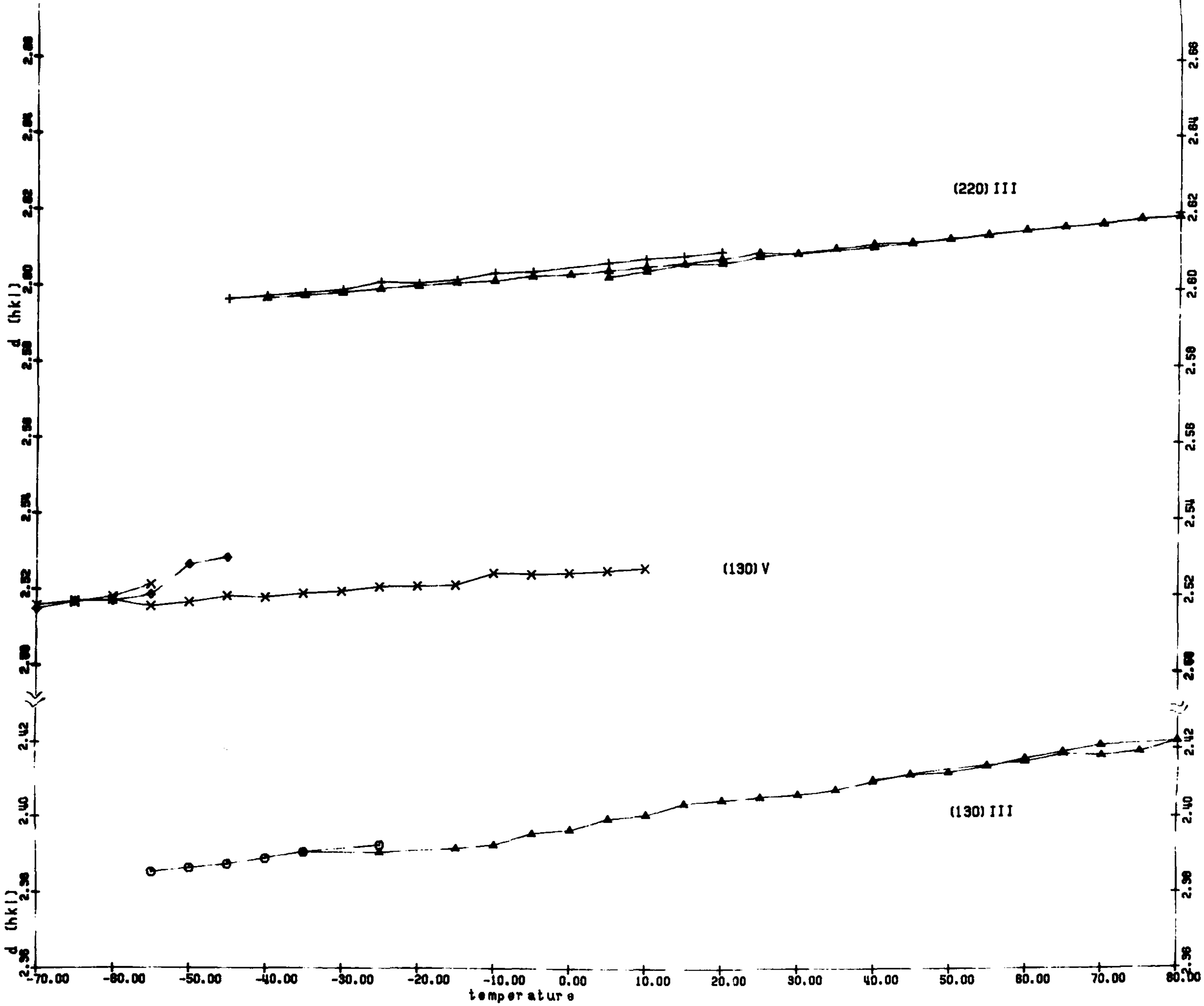



(111) III
(120) III
(210) III
(021) III
(022) V
(211) III
d (hkl)
d (hkl)



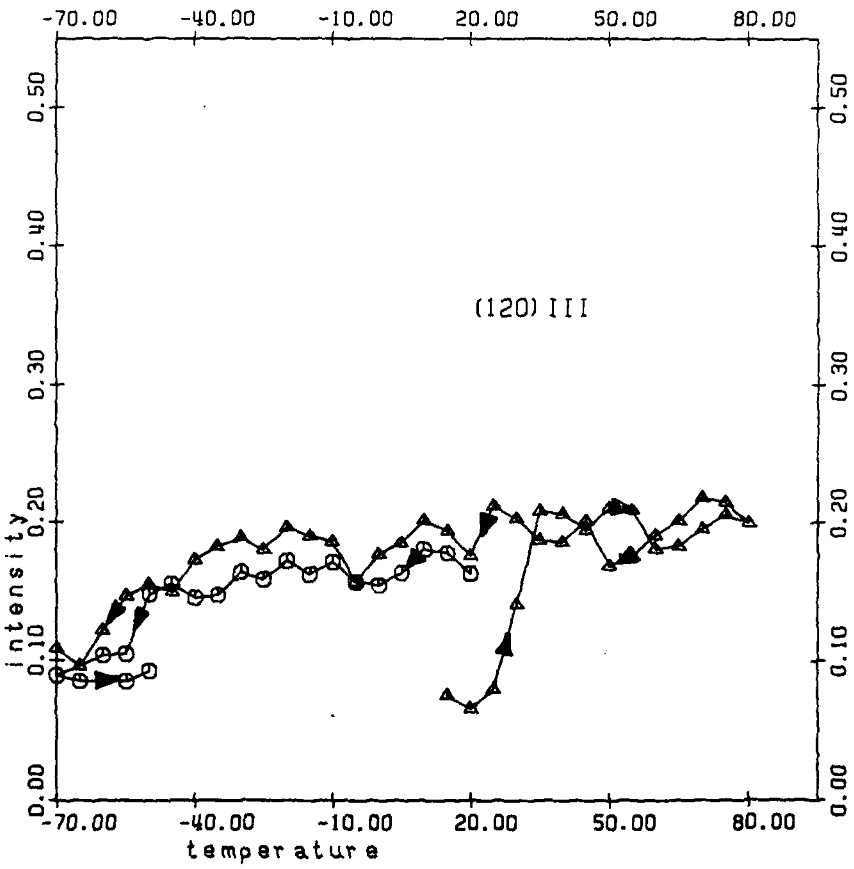
52/2 87
d (hkl)
2.56
2.58
2.60
2.62
2.64
2.66
2.68
2.70
2.72
2.80
2.82
3.08
3.10
3.12
3.14
(220) III
(020) IV
(211) III
(022) V

0806/N50/29752/2.87



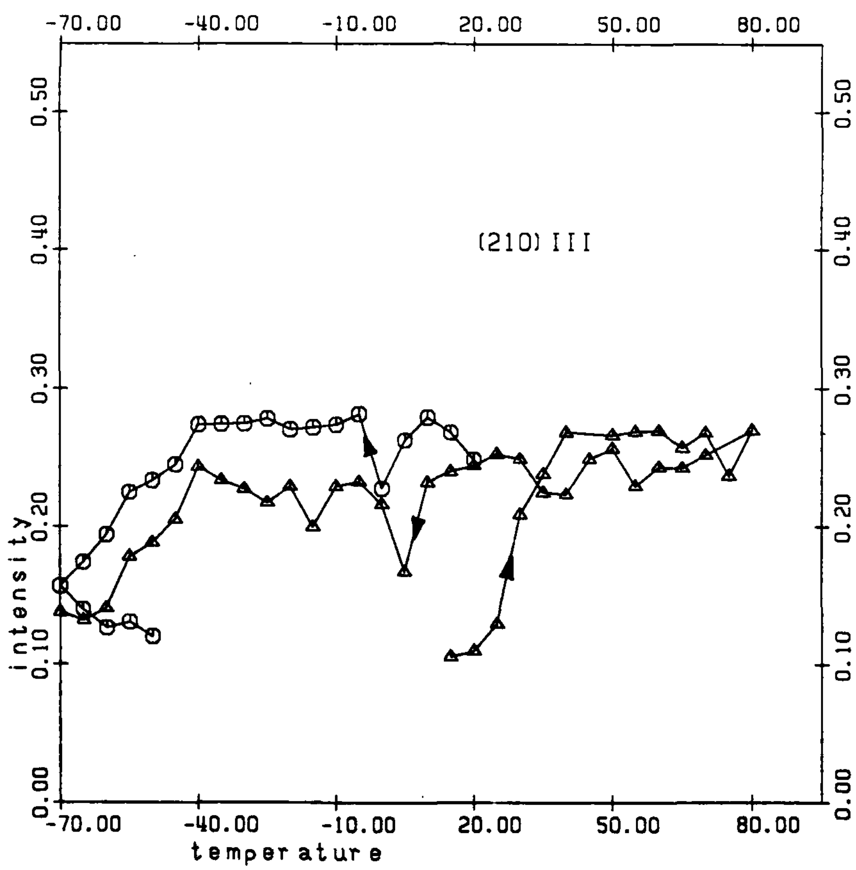
Intensities

kf290988
20/-70/80/-70



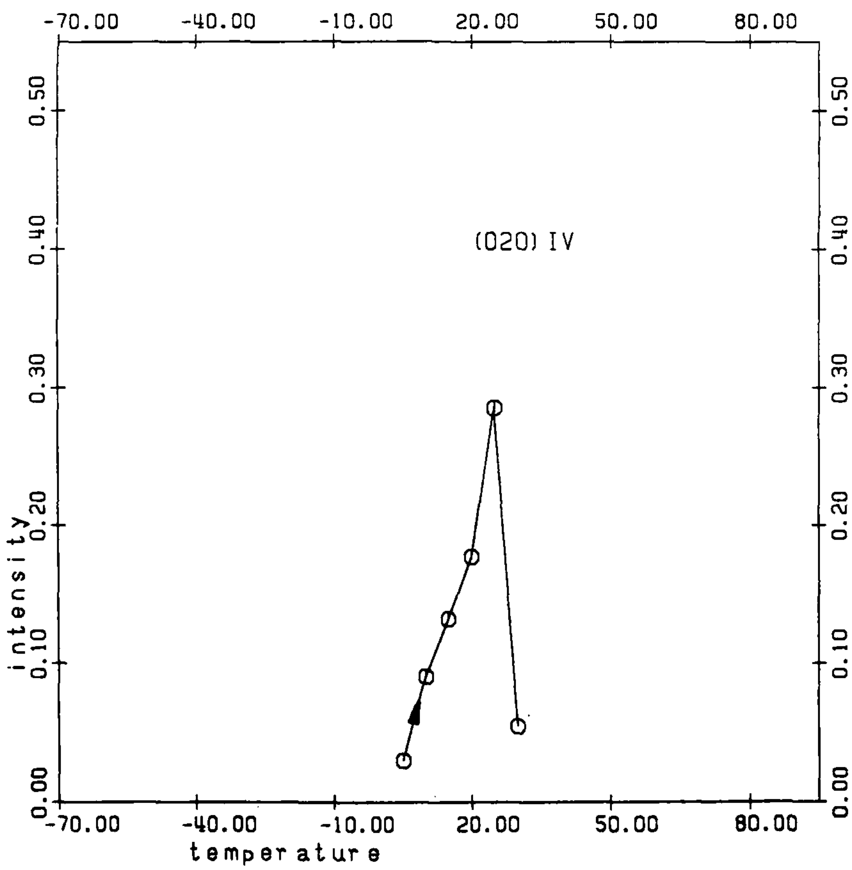
Intensities

kf290988
20/-70/80/-70



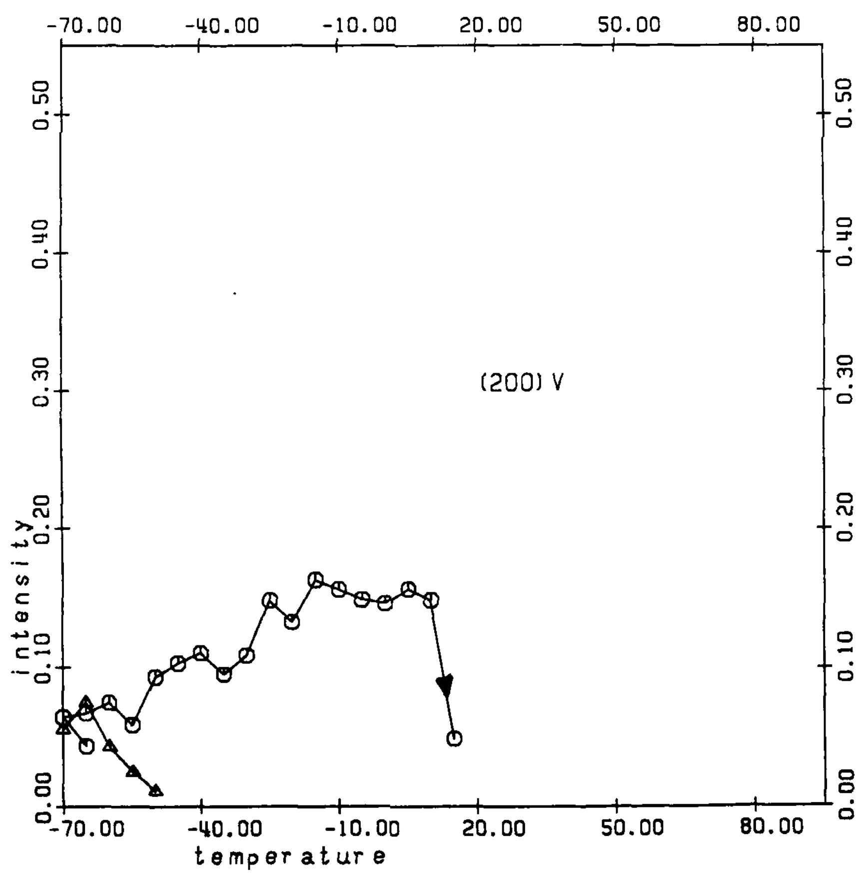
Intensities

kf290988
20/-70/80/-70



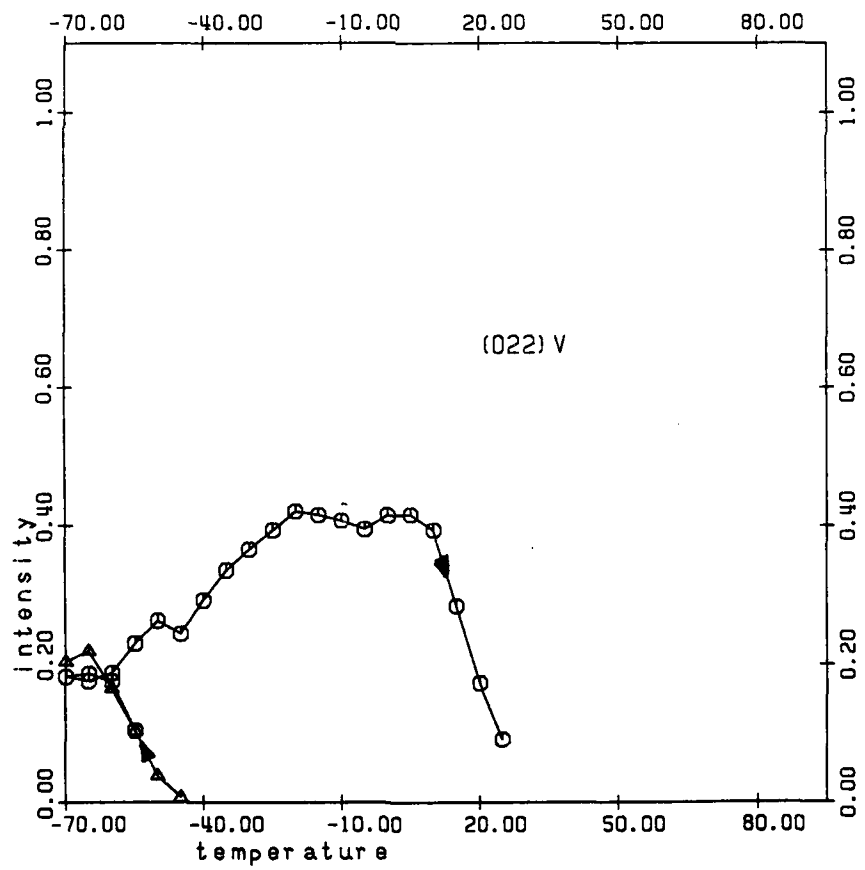
Intensities

kf290988
20/-70/80/-70



Intensities

kf290988
20/-70/80/-70



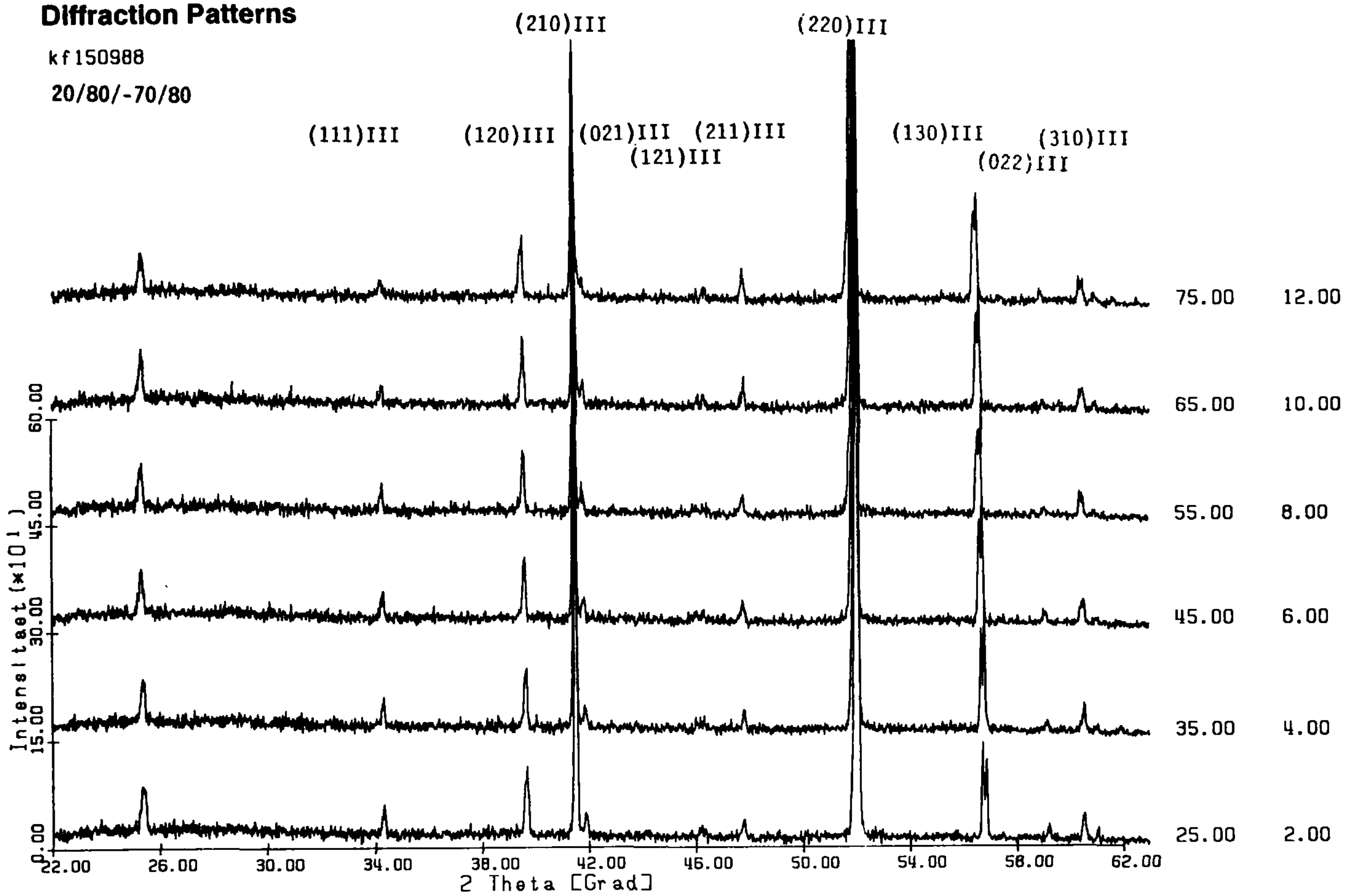
Series
KF 150988
4% KF,humide

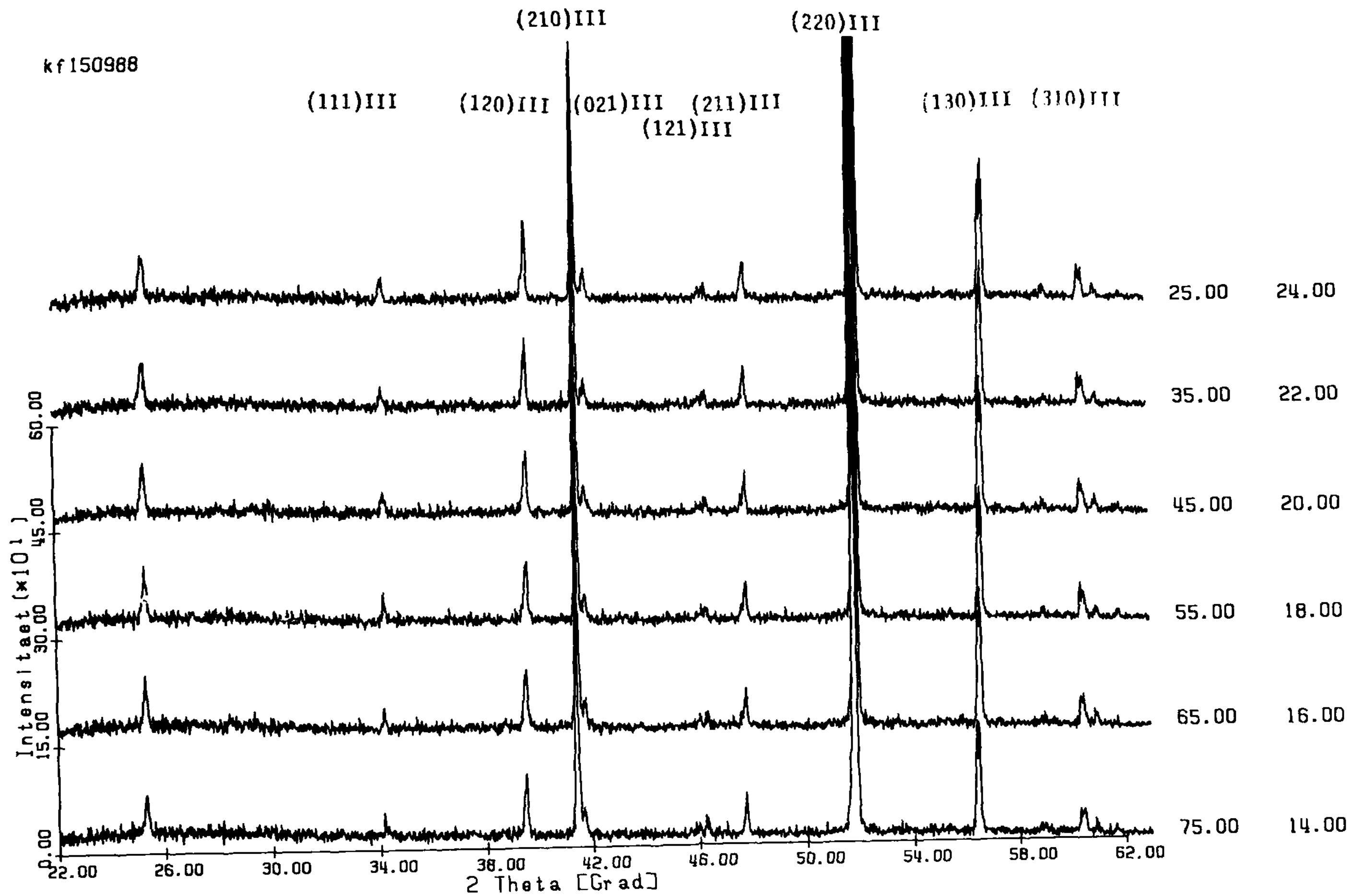
Temperature
Program
20/80/–70/80

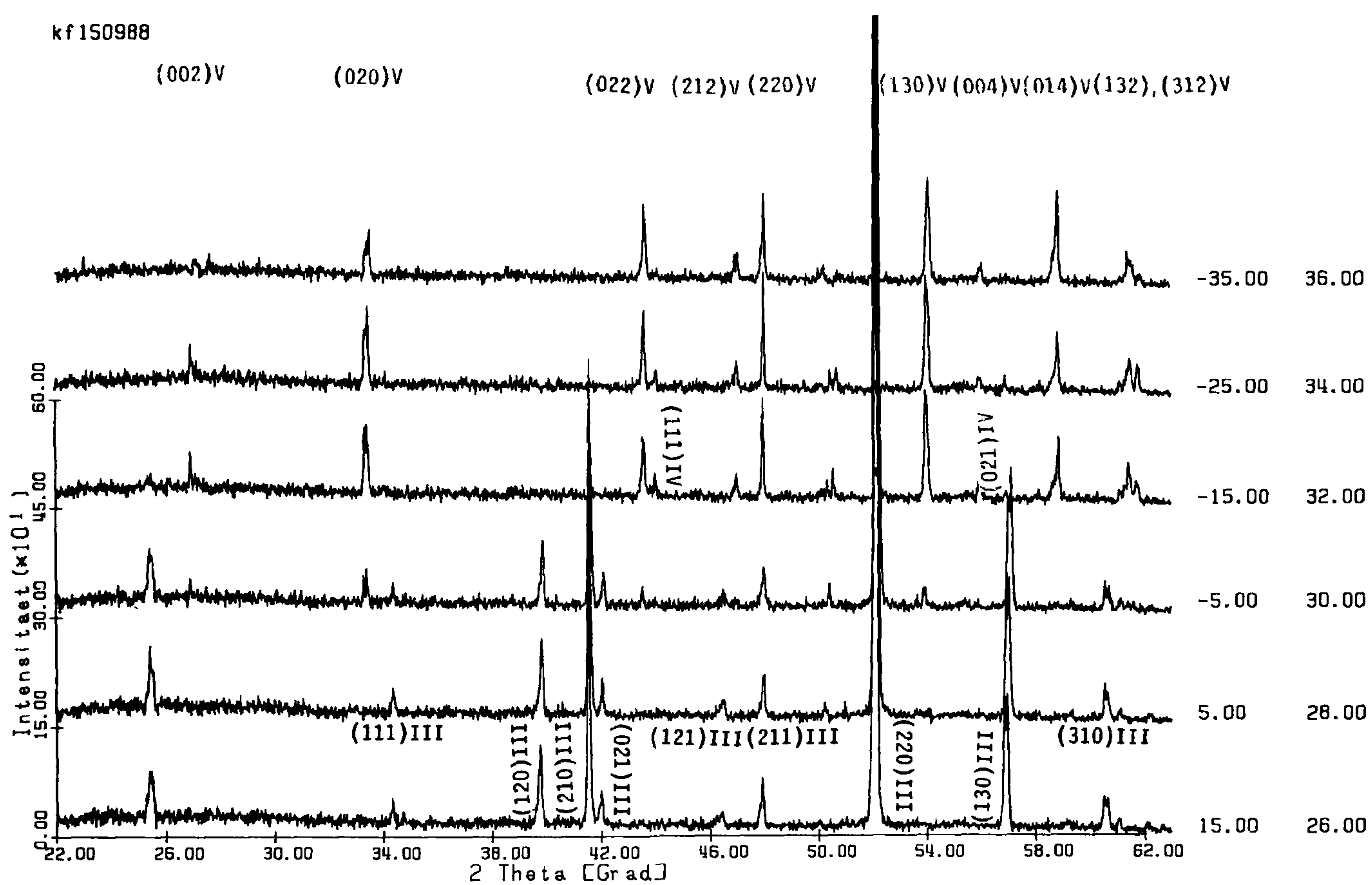
Diffraction Patterns

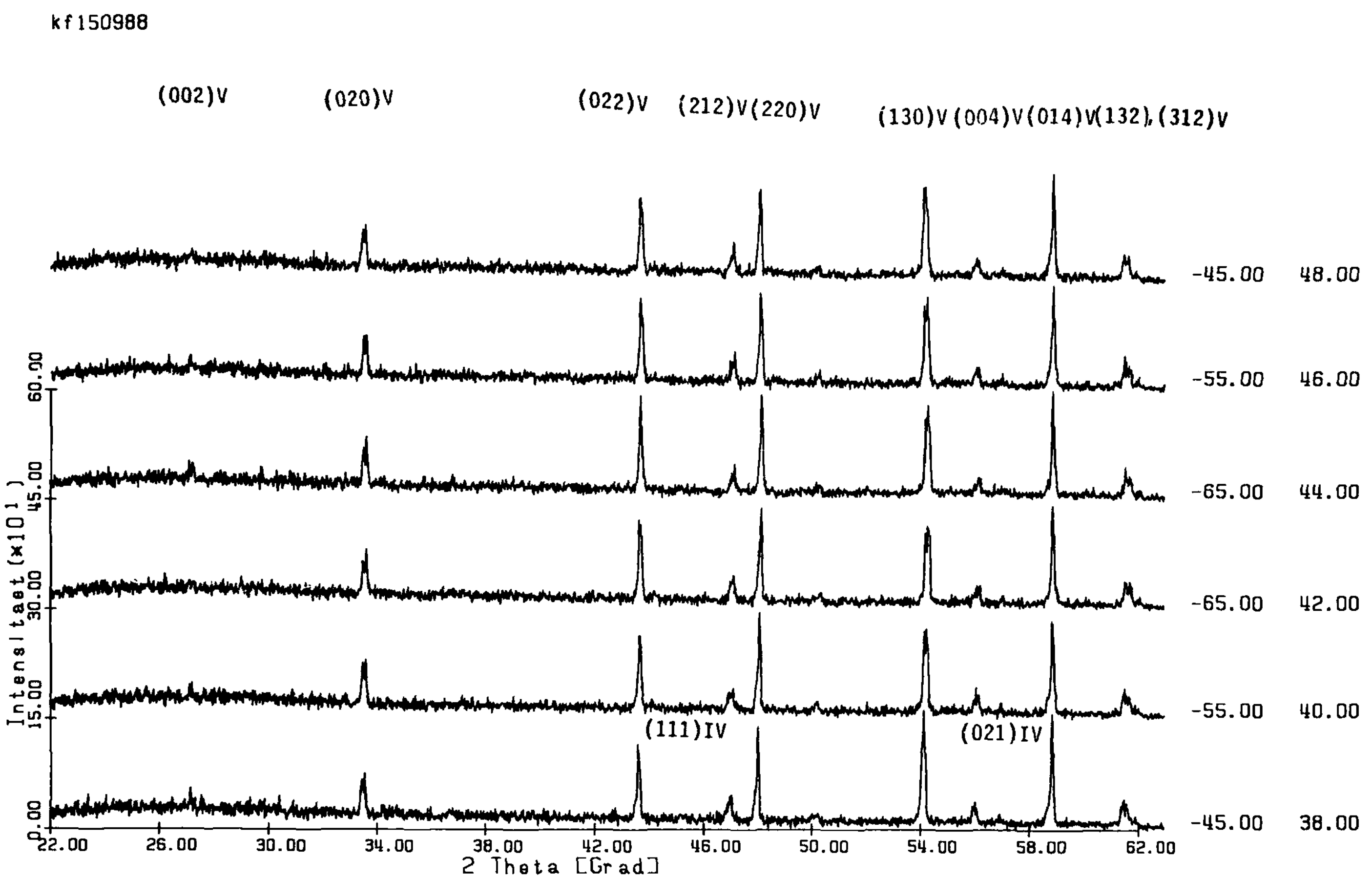
kf150988

20/80/-70/80

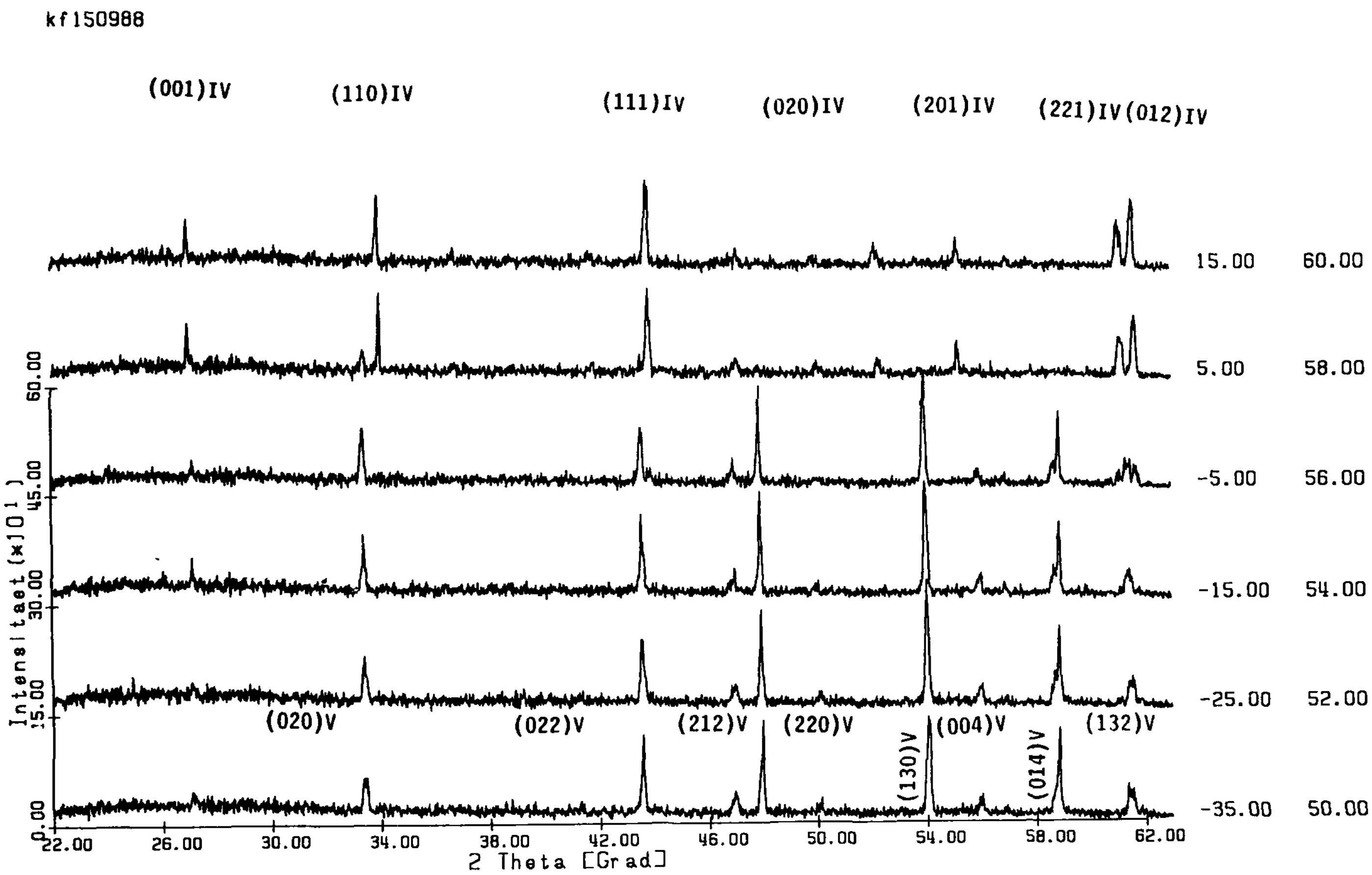



kf150988
(111)III
(120)III
(210)III
(021)III
(121)III
(211)III
(220)III
(130)III
(310)III
25.00 24.00
35.00 22.00
45.00 20.00
55.00 18.00
65.00 16.00
75.00 14.00
Intensitaet (×10¹)
0.00
15.00
30.00
45.00
60.00
22.00
26.00
30.00
34.00
38.00
42.00
46.00
50.00
54.00
58.00
62.00
2 Theta [Grad]

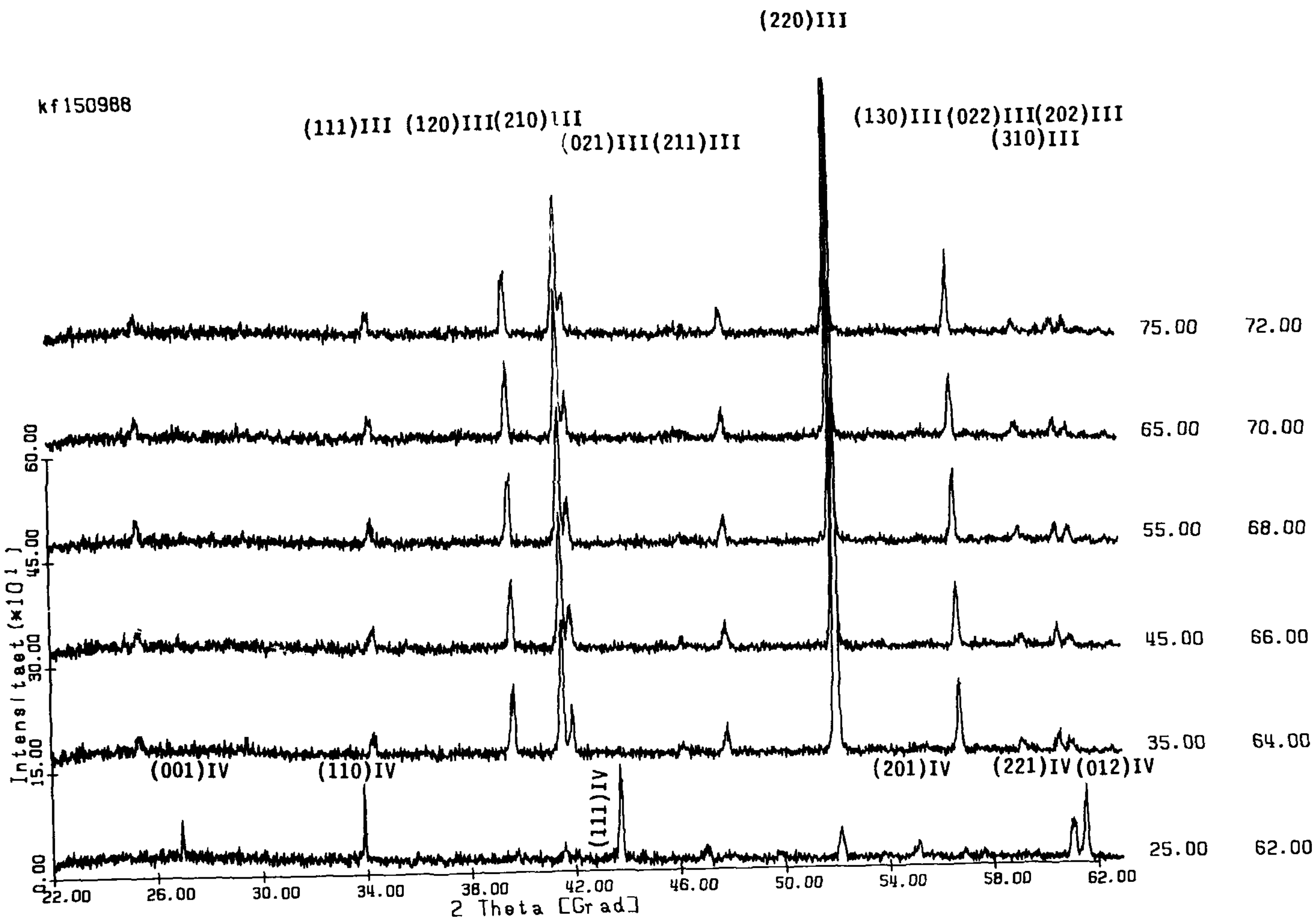

kf150988
(002)V
(020)V
(022)V (212)V (220)V
(130)V (004)V (014)V (132),(312)V
(111)IV
(021)IV
(111)III
(120)III
(210)III
(021)III
(121)III (211)III
(220)III
(130)III
(310)III
-35.00 36.00
-25.00 34.00
-15.00 32.00
-5.00 30.00
5.00 28.00
15.00 26.00
Intensitaet (x10^1)
0.00
15.00
30.00
45.00
60.00
22.00
26.00
30.00
34.00
38.00
42.00
46.00
50.00
54.00
58.00
62.00
2 Theta [Grad]



kf150988
(002)V
(020)V
(022)V
(212)V(220)V
(130)V(004)V(014)V(132),(312)V
(111)IV
(021)IV
-45.00 48.00
-55.00 46.00
-65.00 44.00
-65.00 42.00
-55.00 40.00
-45.00 38.00
Intensitaet (x10¹)
0.00
15.00
30.00
45.00
60.00
22.00
26.00
30.00
34.00
38.00
42.00
46.00
50.00
54.00
58.00
62.00
2 Theta [Grad]



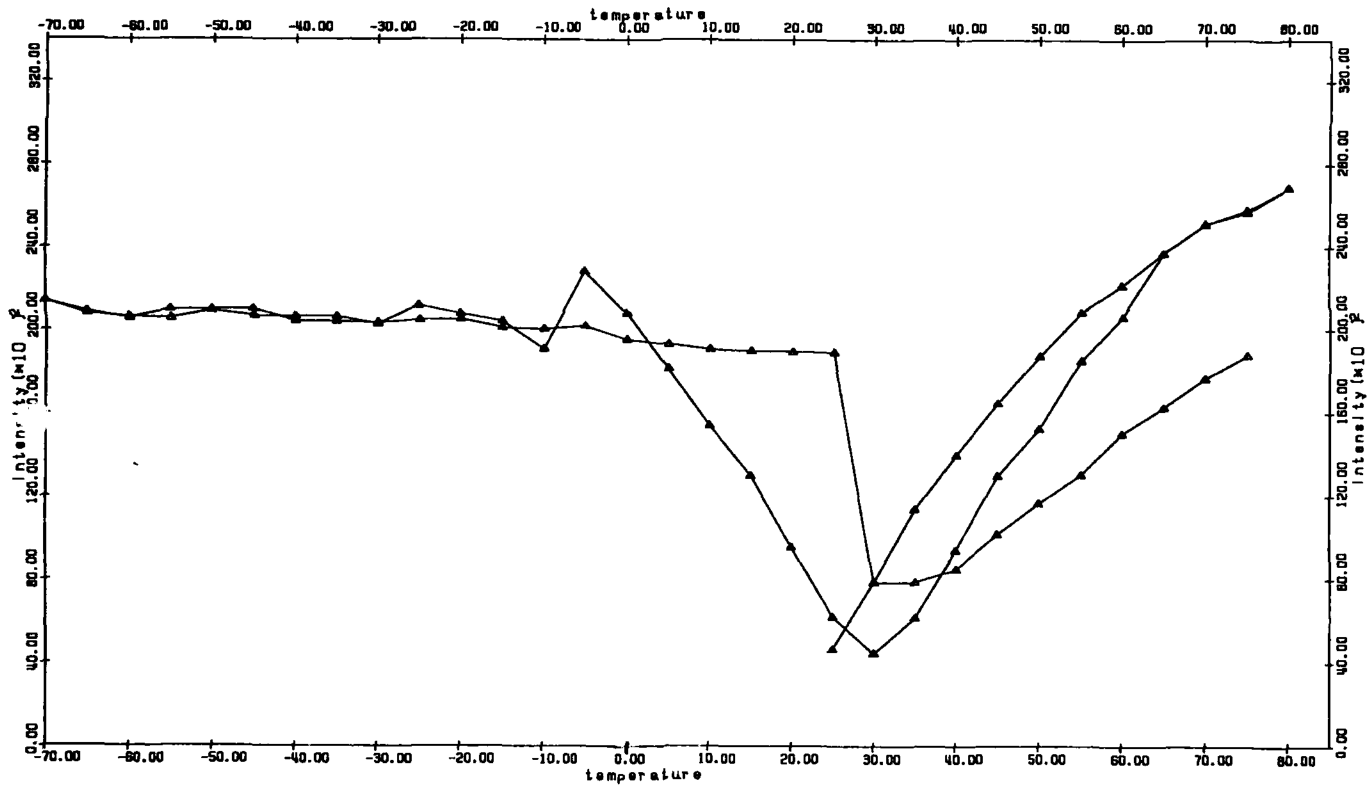
kf150988
(001)IV
(110)IV
(111)IV
(020)IV
(201)IV
(221)IV
(012)IV
15.00
60.00
5.00
58.00
-5.00
56.00
-15.00
54.00
-25.00
52.00
-35.00
50.00
(020)V
(022)V
(212)V
(220)V
(130)V
(004)V
(014)V
(132)V
Intensitaet (x10^1)
0.00
15.00
30.00
45.00
60.00
22.00
26.00
30.00
34.00
38.00
42.00
46.00
50.00
54.00
58.00
62.00
2 Theta [Grad]



kf150988
(220)III
(111)III (120)III(210)III
(021)III(211)III
(130)III(022)III(202)III
(310)III
(001)IV
(110)IV
(111)IV
(201)IV
(221)IV(012)IV
75.00 72.00
65.00 70.00
55.00 68.00
45.00 66.00
35.00 64.00
25.00 62.00
Intensitaet (*10¹)
0.00
15.00
30.00
45.00
60.00
22.00
26.00
30.00
34.00
38.00
42.00
46.00
50.00
54.00
58.00
62.00
2 Theta [Grad]

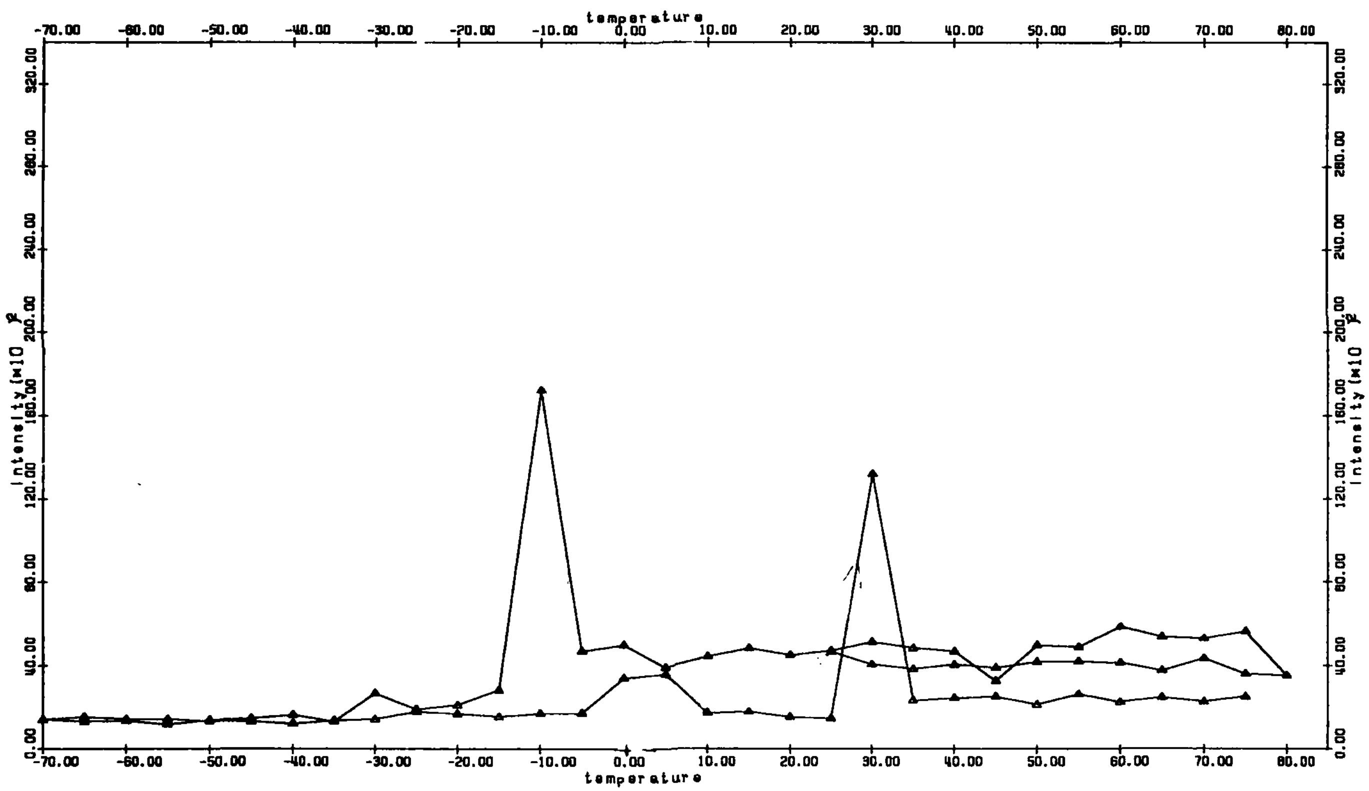
Difference Curve Y(T)

KF150988
20/80/-70/80



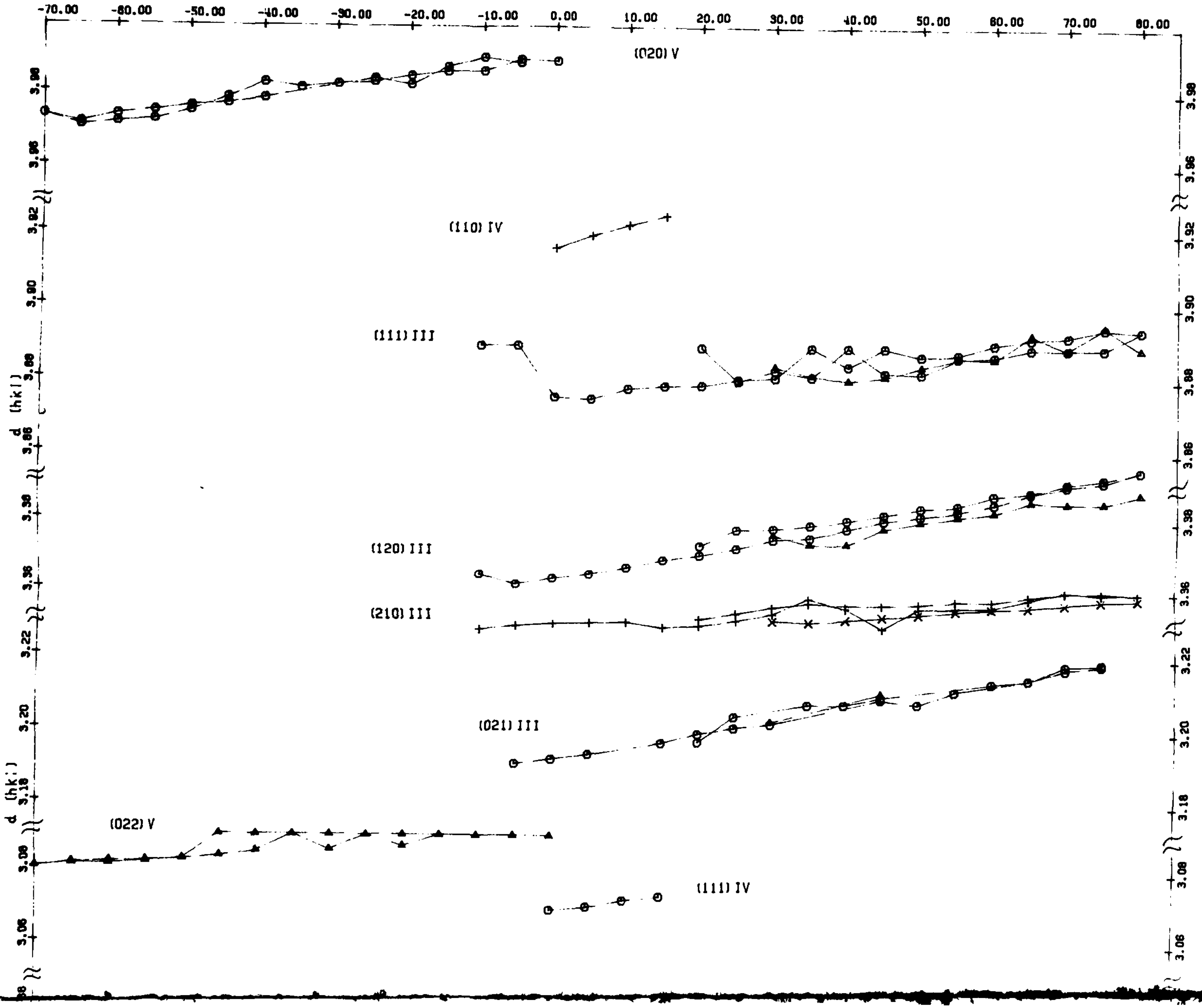
Difference Curve dy(T)

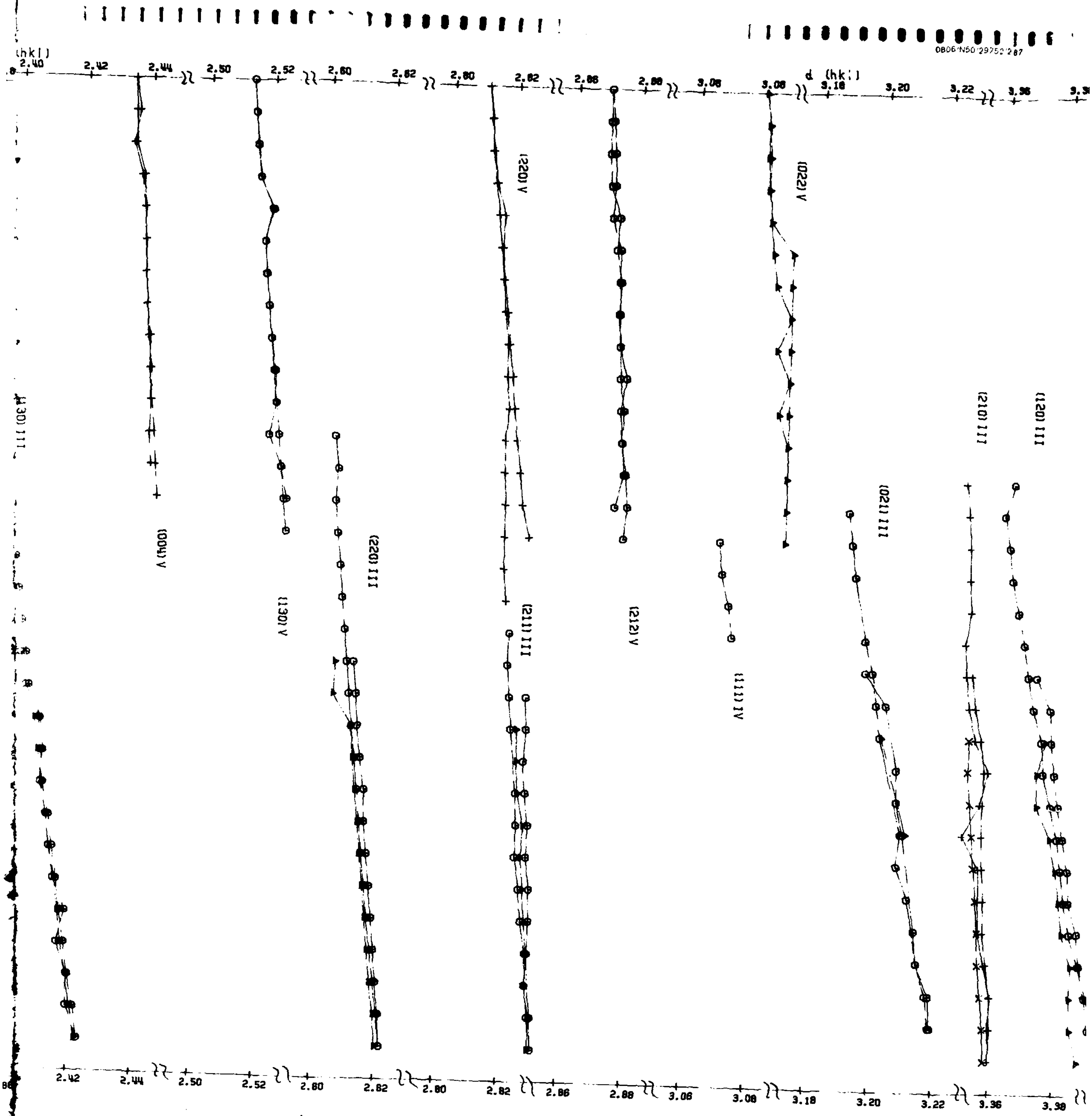
KF150988
20/80/-70/80



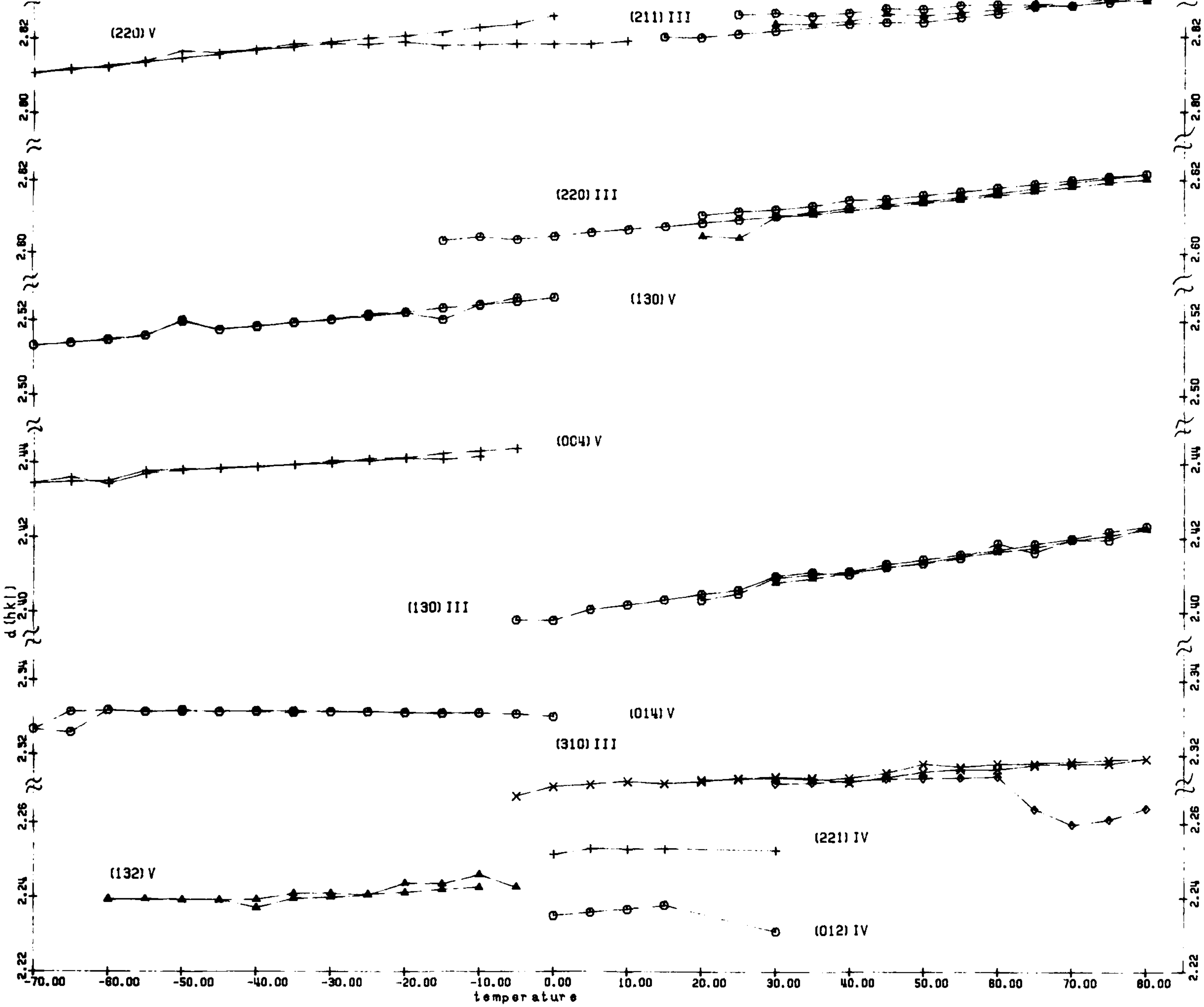
Lattice Plane Distances

kf150988
20/80/-70/80





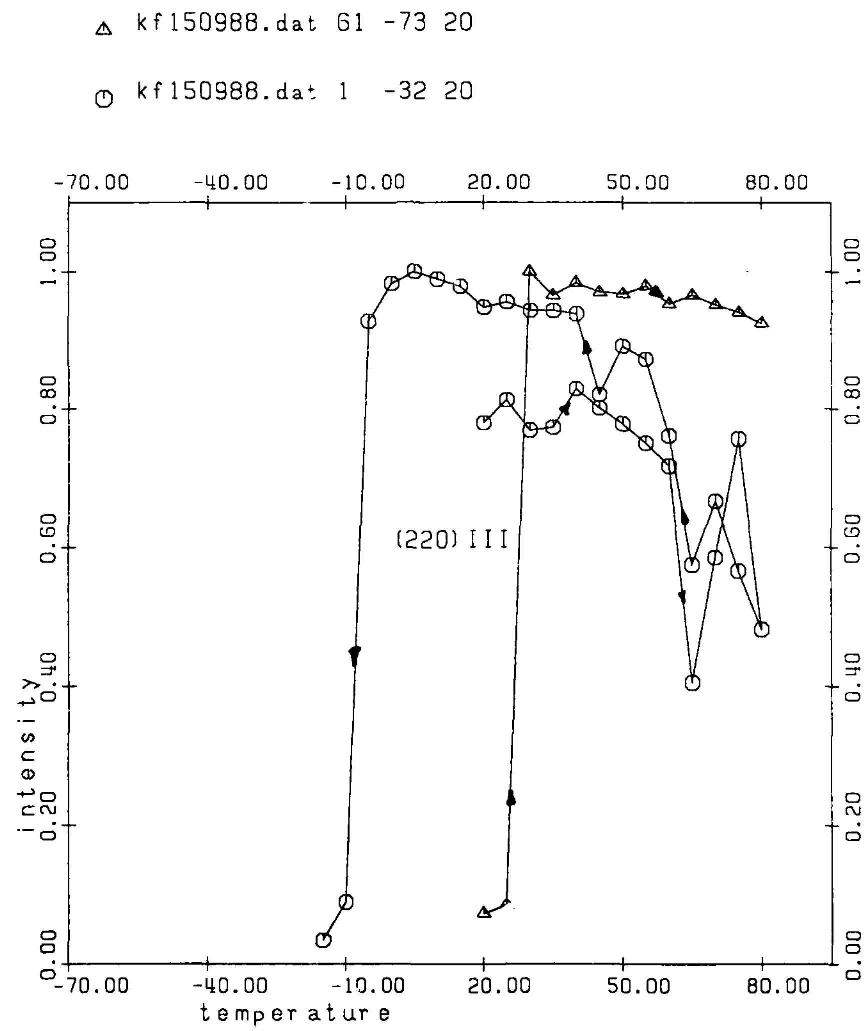
d (hk l)
(130) III
(004) V
(130) V
(220) III
(220) V
(211) III
(212) V
(111) IV
(022) V
(021) III
(210) III
(120) III
0806/N50/29752/287



(220) V
(211) III
(220) III
(130) V
(004) V
(130) III
(014) V
(310) III
(221) IV
(132) V
(012) IV
d (hkl)
temperature

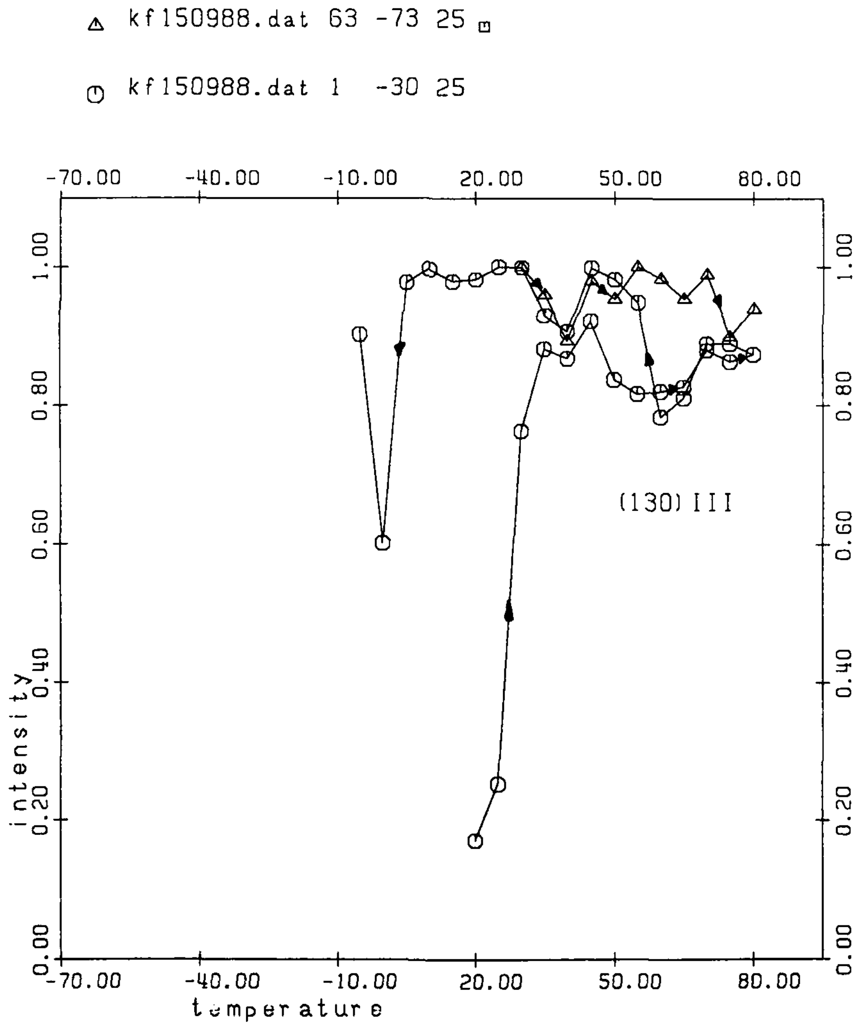
Intensities

KF150988
20/80/-70/80



Intensities

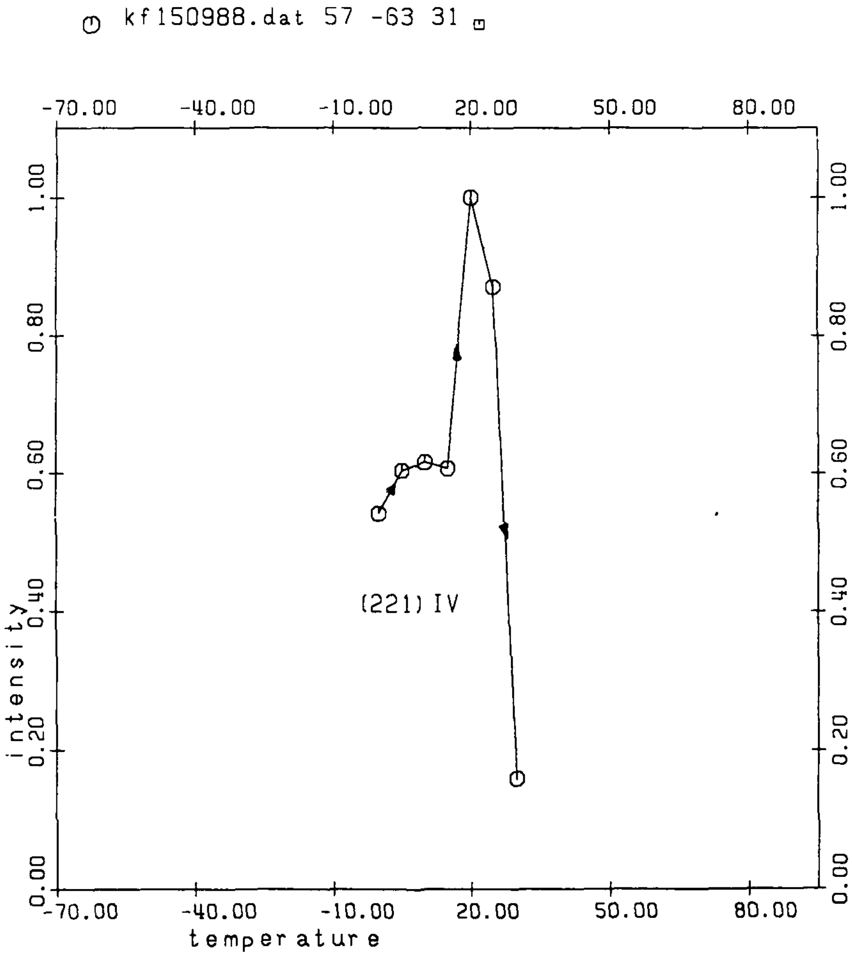
KF150988
20/80/-70/80



Intensities

KF150988

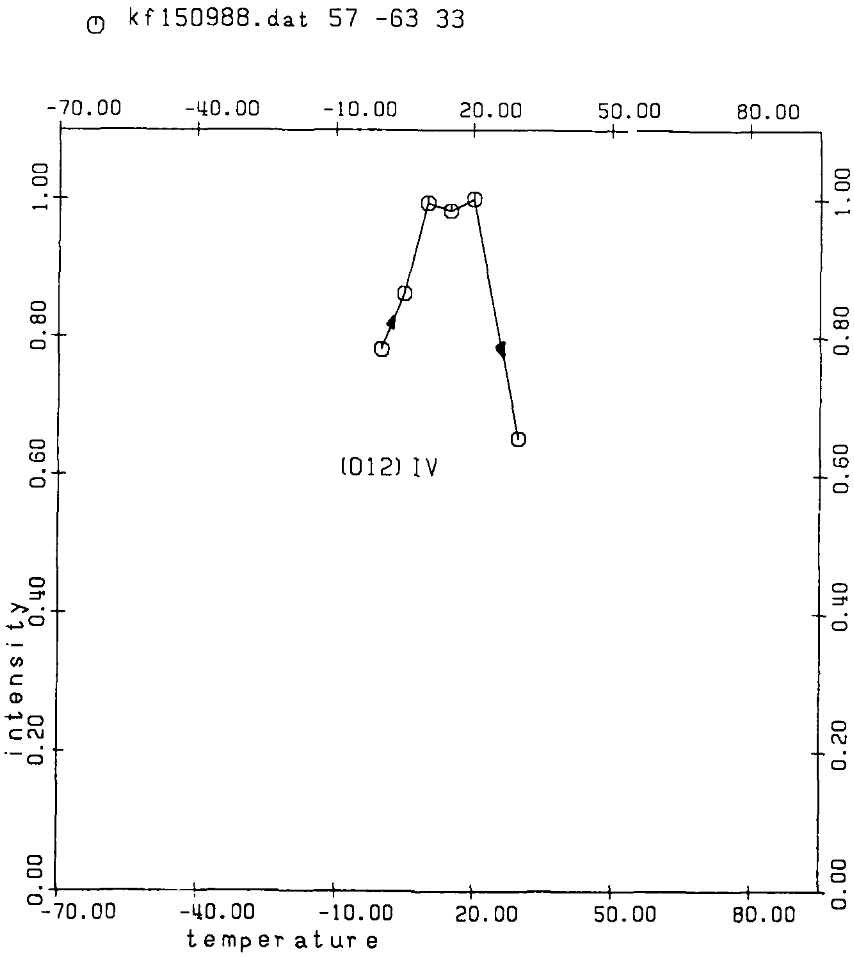
20/80/-70/80



Intensities

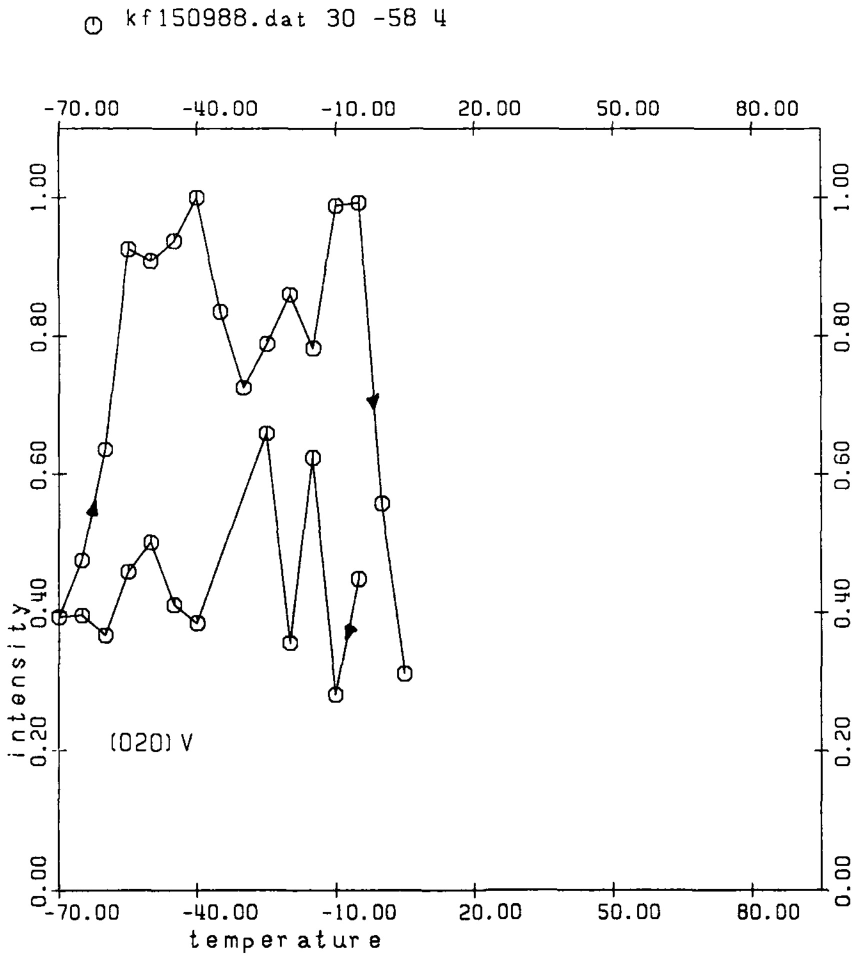
KF150988

20/80/-70/80



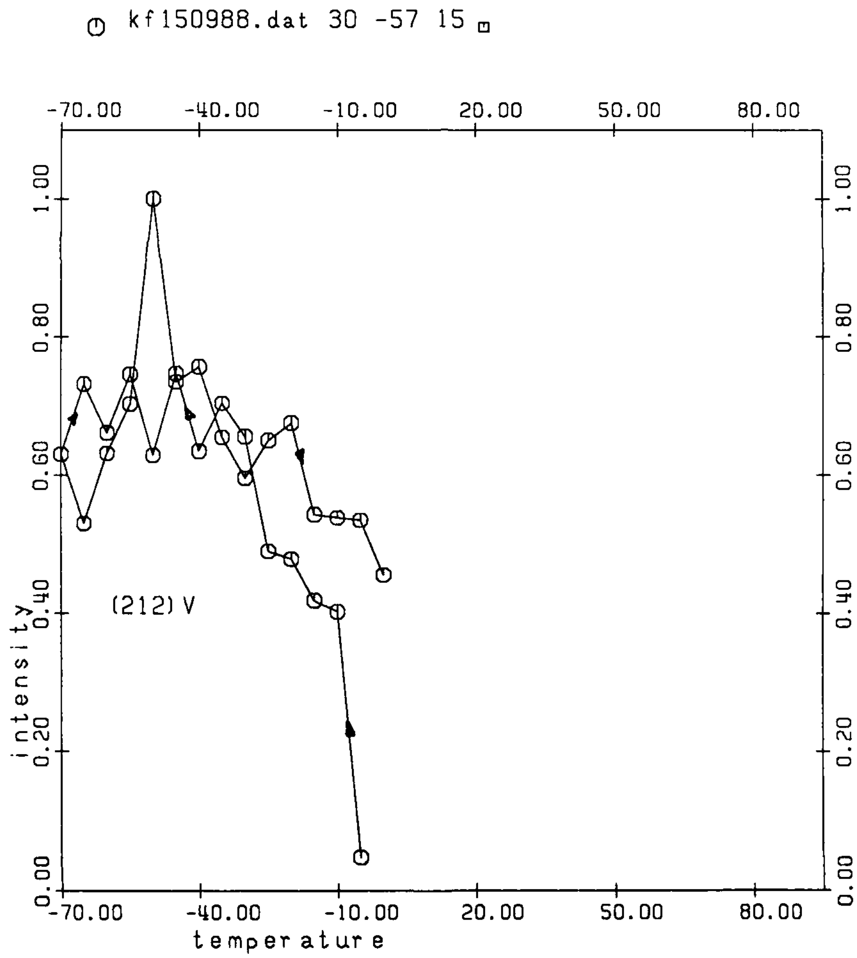
Intensities

KF150988
20/80/-70/80



Intensities

KF150988
20/80/-70/80



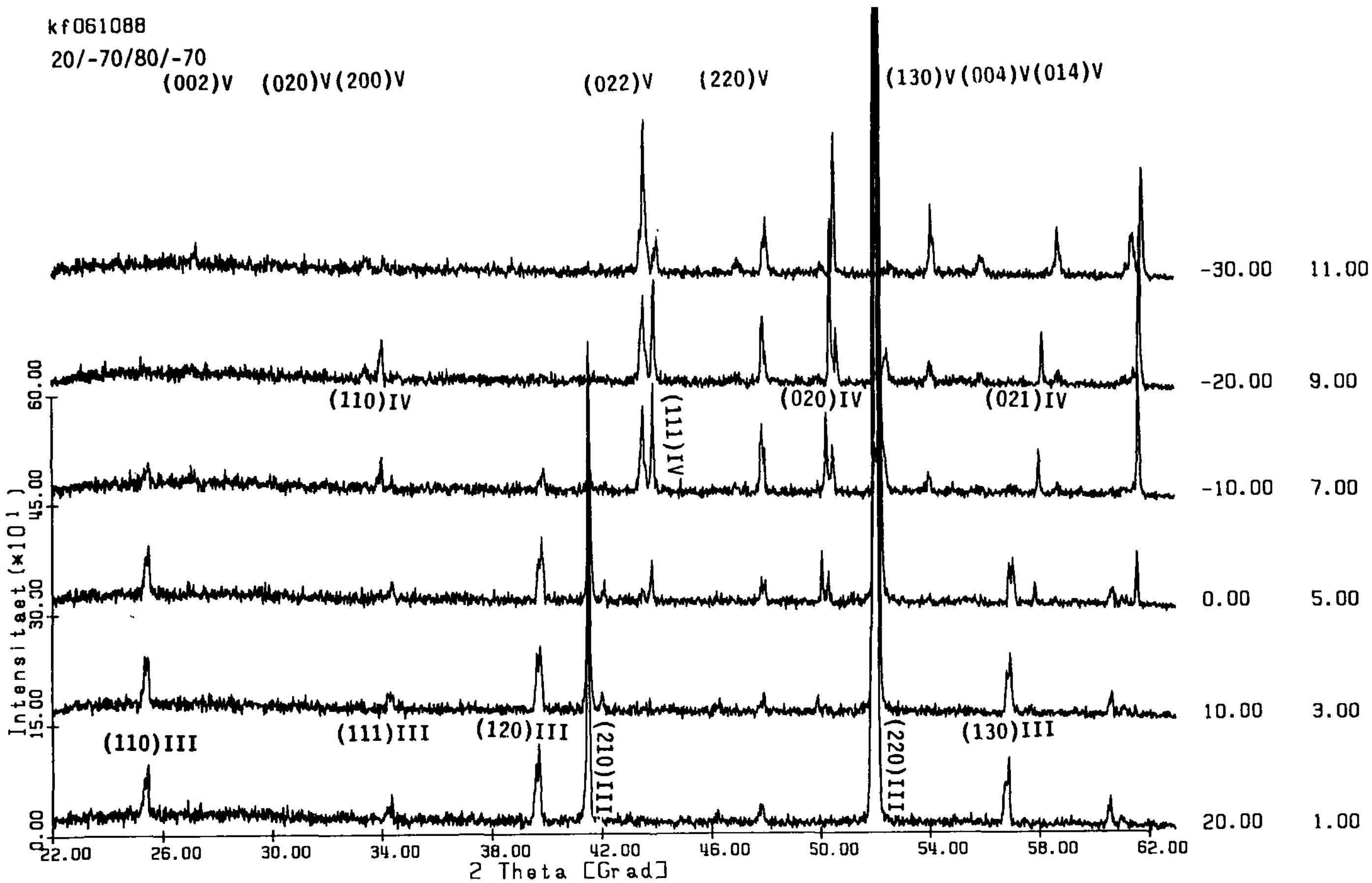
Series
KF 061088
4% KF,humide

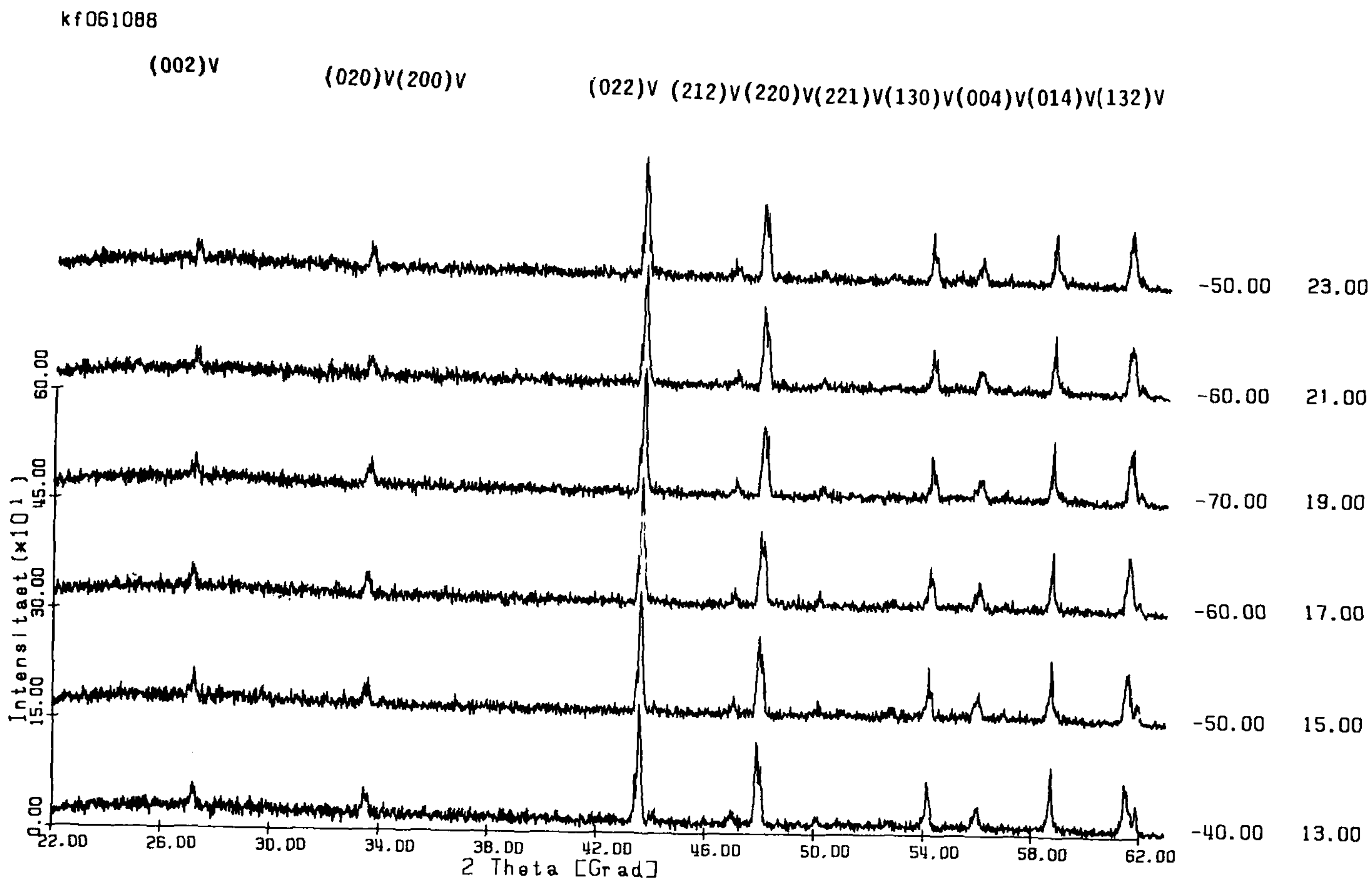
Temperature
Program
20/−70/80/−70

Diffraction Patterns

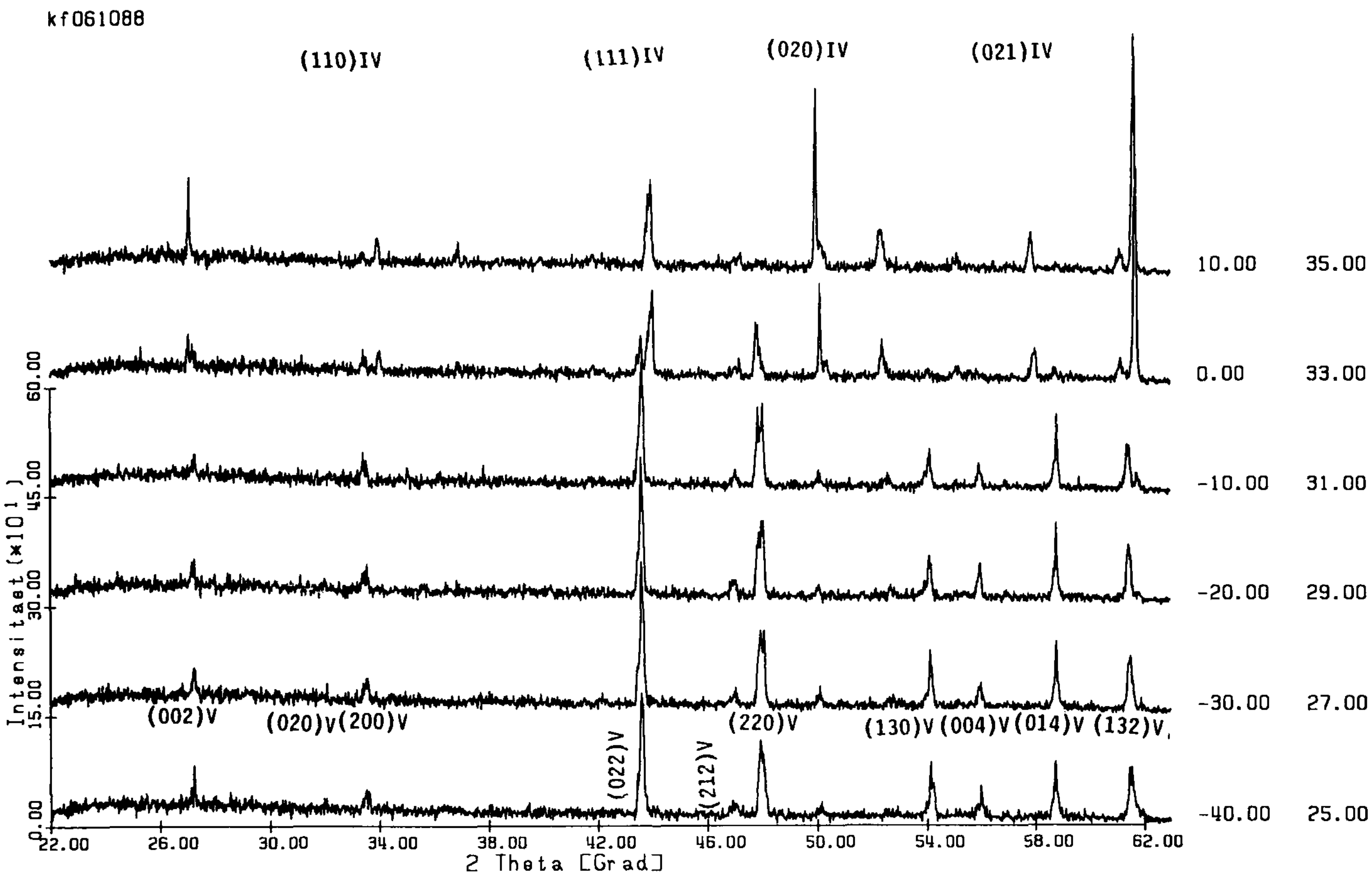
kf061088

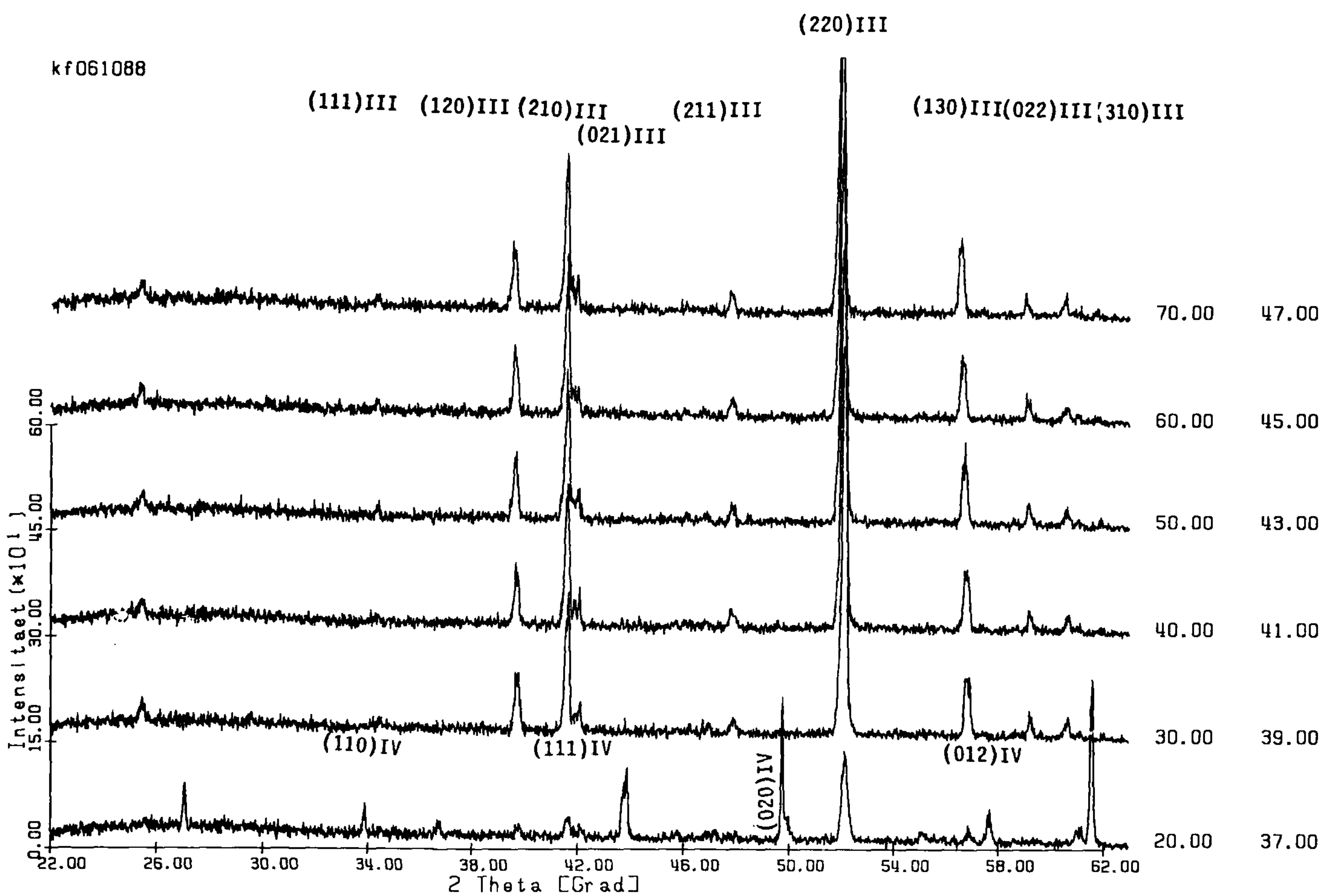
20/-70/80/-70

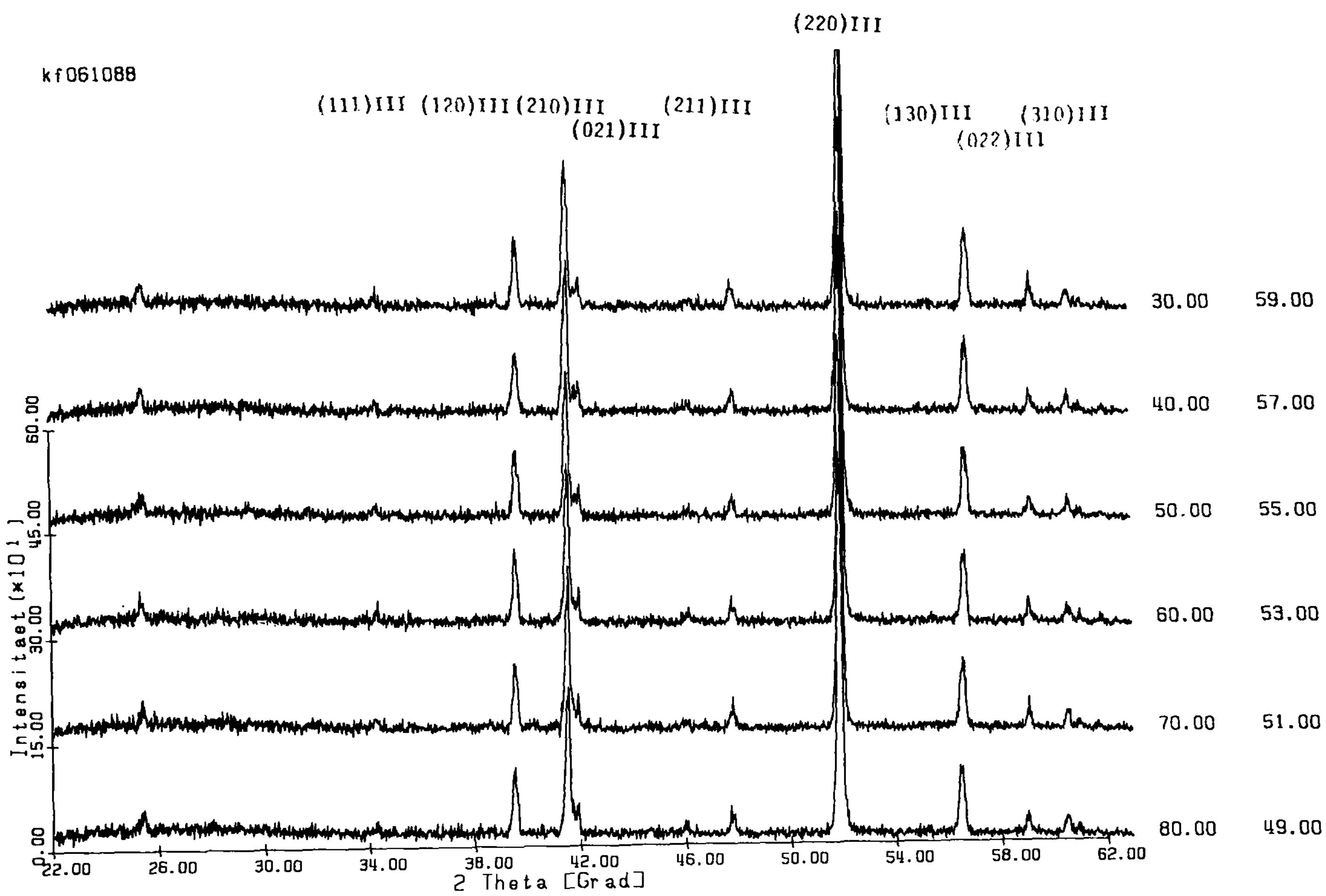


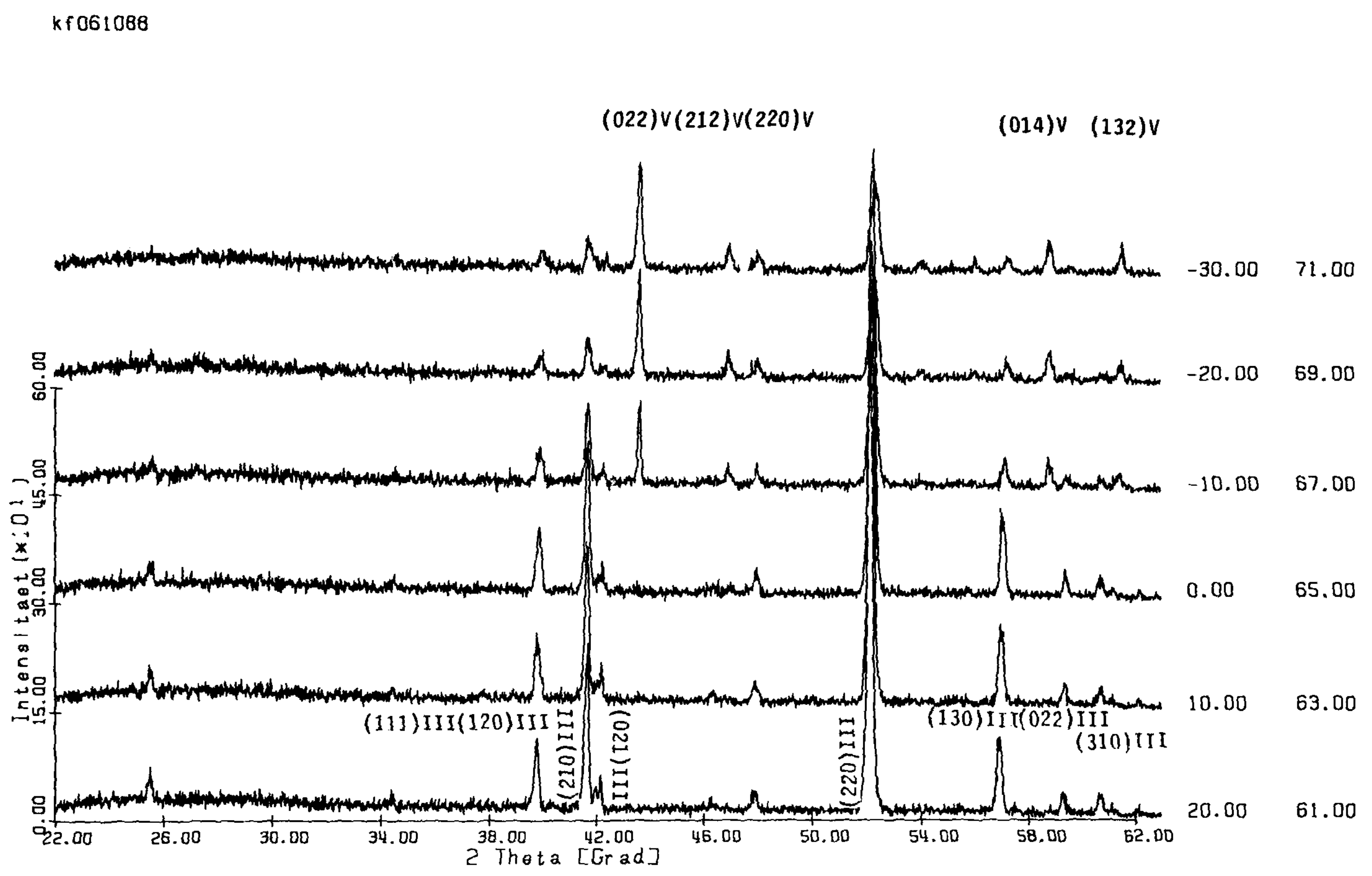


kf061088
(002)V
(020)V(200)V
(022)V (212)V(220)V(221)V(130)V(004)V(014)V(132)V
-50.00 23.00
-60.00 21.00
-70.00 19.00
-60.00 17.00
-50.00 15.00
-40.00 13.00
Intensitaet (x10^1)
0.00
15.00
30.00
45.00
60.00
22.00
26.00
30.00
34.00
38.00
42.00
46.00
50.00
54.00
58.00
62.00
2 Theta [Grad]

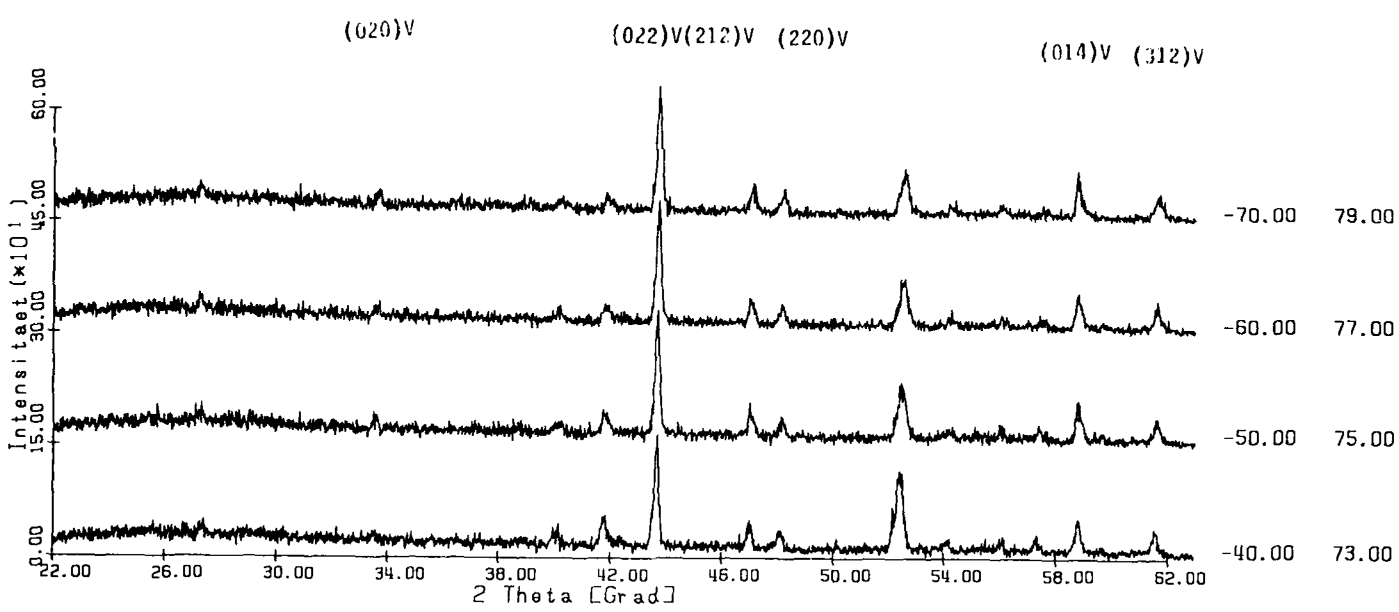

kf061088
(110)IV
(111)IV
(020)IV
(021)IV
10.00 35.00
0.00 33.00
-10.00 31.00
-20.00 29.00
-30.00 27.00
-40.00 25.00
(002)V
(020)V(200)V
(022)V
(212)V
(220)V
(130)V (004)V (014)V (132)V
Intensitaet (*10 1)
0.00
15.00
30.00
45.00
60.00
22.00
26.00
30.00
34.00
38.00
42.00
46.00
50.00
54.00
58.00
62.00
2 Theta [Grad]


kf061088
(220)III
(111)III
(120)III
(210)III
(021)III
(211)III
(130)III
(022)III
(310)III
(110)IV
(111)IV
(020)IV
(012)IV
70.00
60.00
50.00
40.00
30.00
20.00
47.00
45.00
43.00
41.00
39.00
37.00
Intensitaet (x10[1])
0.00
15.00
30.00
45.00
60.00
22.00
26.00
30.00
34.00
38.00
42.00
46.00
50.00
54.00
58.00
62.00
2 Theta [Grad]


kf061088
(111)III
(120)III
(210)III
(021)III
(211)III
(220)III
(130)III
(022)III
(310)III
30.00 59.00
40.00 57.00
50.00 55.00
60.00 53.00
70.00 51.00
80.00 49.00
Intensitaet (x10^1)
0.00
15.00
30.00
45.00
60.00
22.00
26.00
30.00
34.00
38.00
42.00
46.00
50.00
54.00
58.00
62.00
2 Theta [Grad]


kf061088
(022)V(212)V(220)V
(014)V (132)V
-30.00 71.00
-20.00 69.00
-10.00 67.00
0.00 65.00
10.00 63.00
20.00 61.00
(111)III(120)III
(210)III
(021)III
(220)III
(130)III(022)III
(310)III
Intensitaet (x10¹)
0.00
15.00
30.00
45.00
60.00
22.00
26.00
30.00
34.00
38.00
42.00
46.00
50.00
54.00
58.00
62.00
2 Theta [Grad]

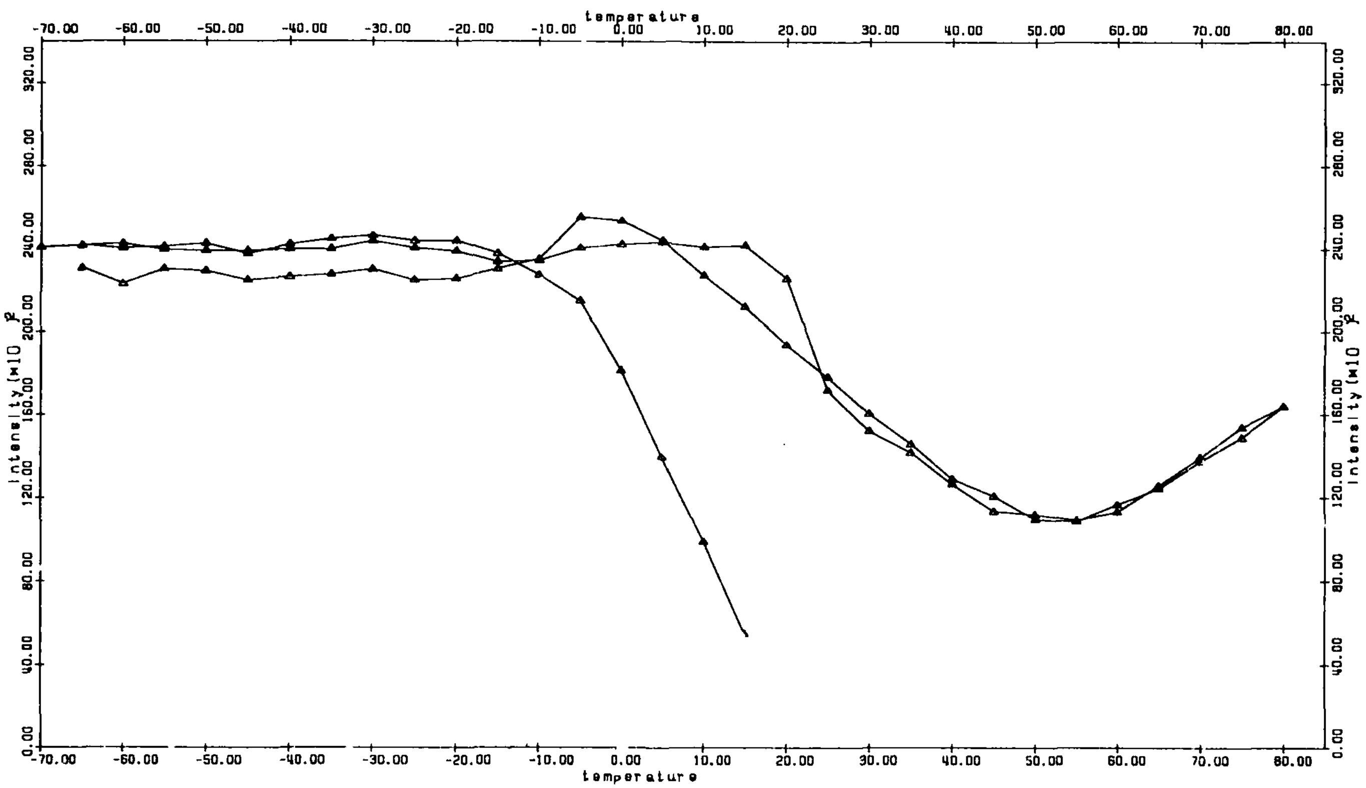
kf061088



(020)V
(022)V(212)V
(220)V
(014)V
(312)V
Intensitaet (*10[1])
60.00
45.00
30.00
15.00
0.00
22.00
26.00
30.00
34.00
38.00
42.00
46.00
50.00
54.00
58.00
62.00
2 Theta [Grad]
-70.00
79.00
-60.00
77.00
-50.00
75.00
-40.00
73.00

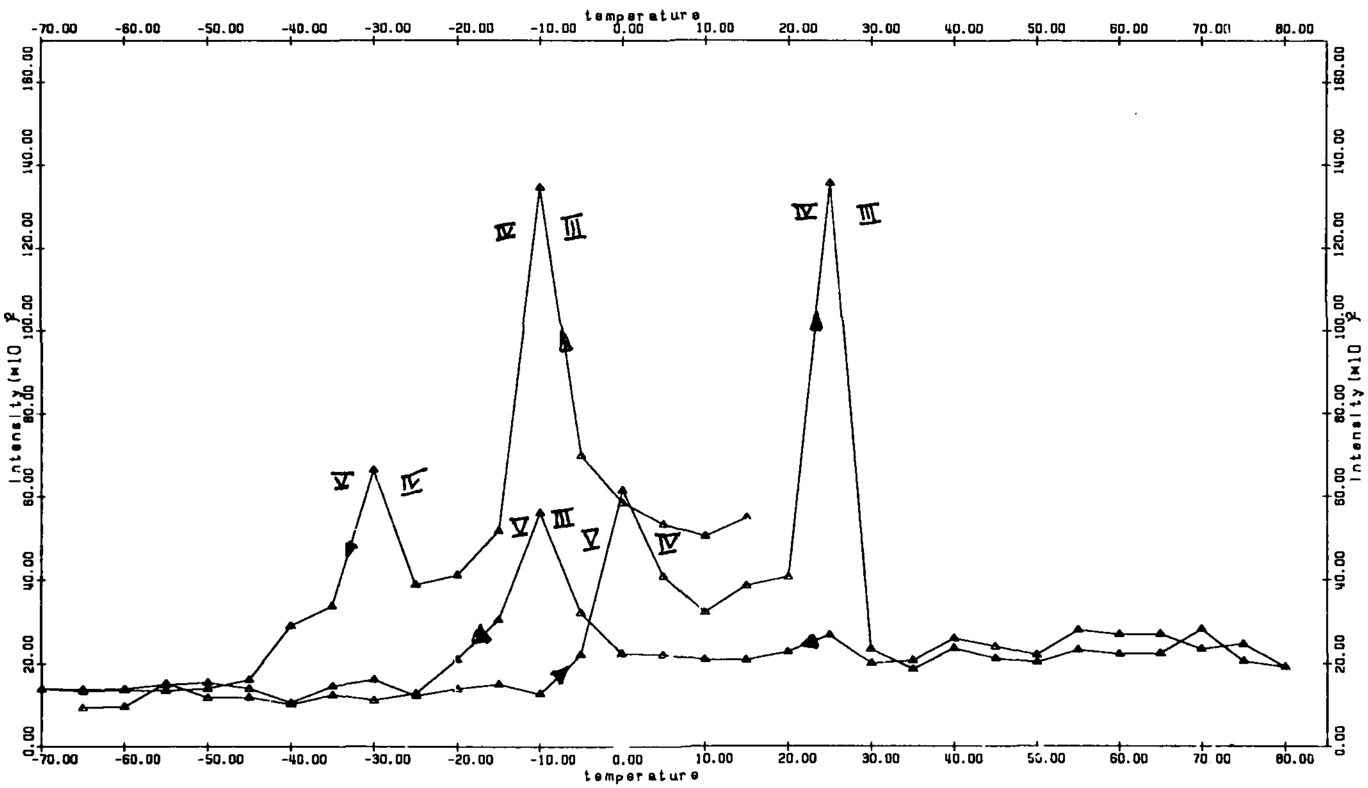
Difference Curve Y(T)

KF061088
20/-70/80/-70



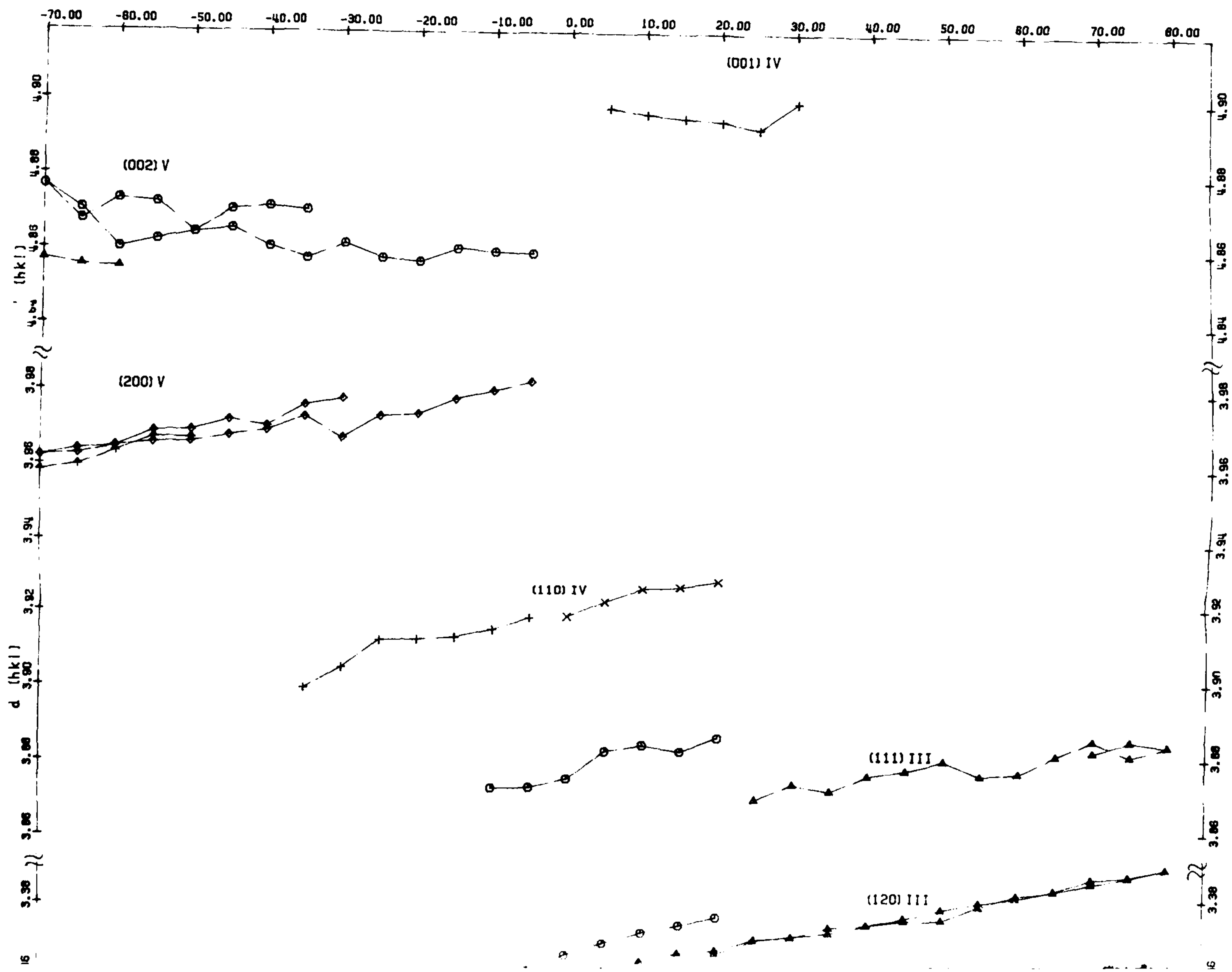
Difference Curve dy(T)

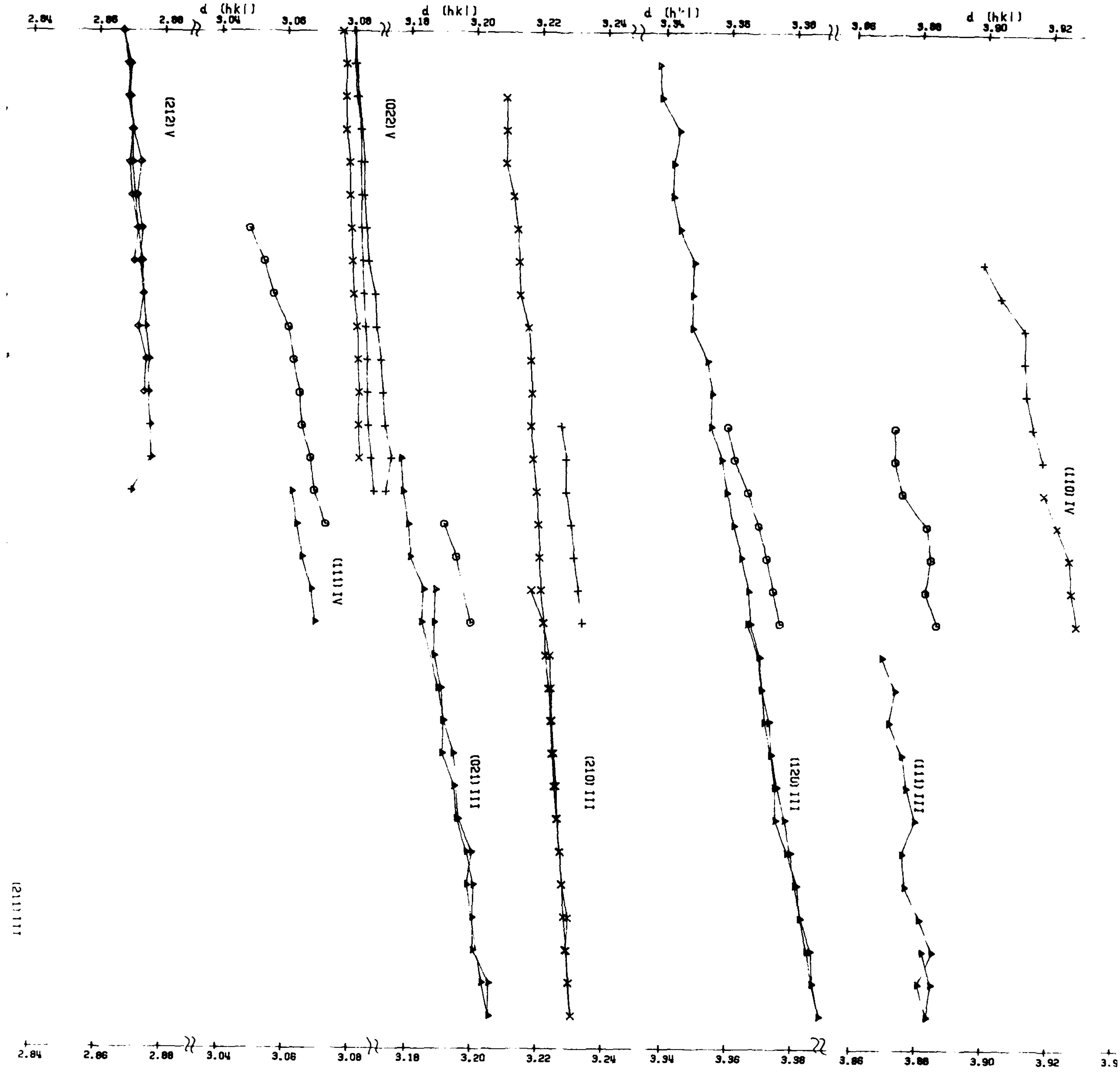
KF061088
20/-70/80/-70



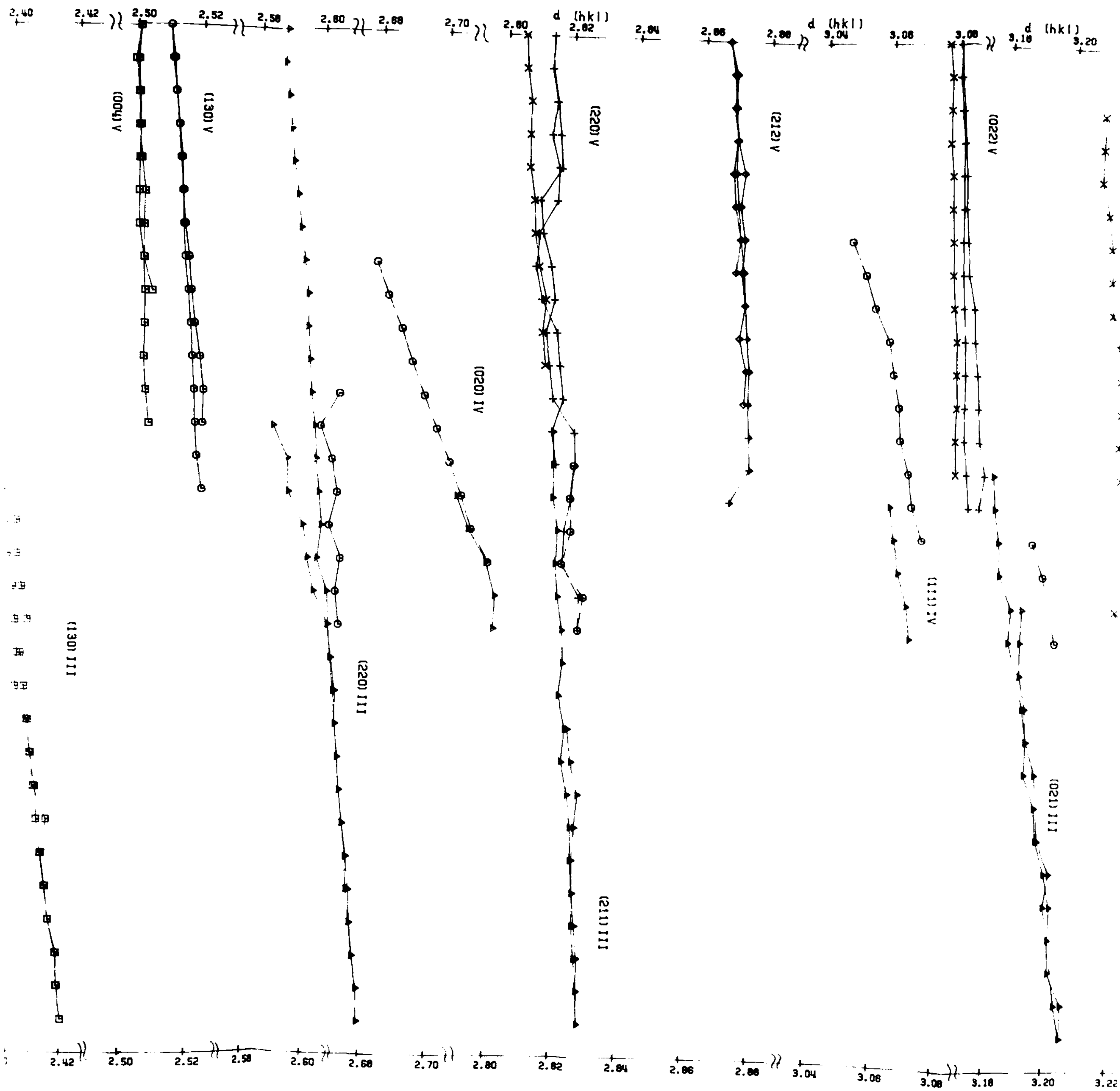
Lattice Plane Distances

kf061088
20/-70/80/-70

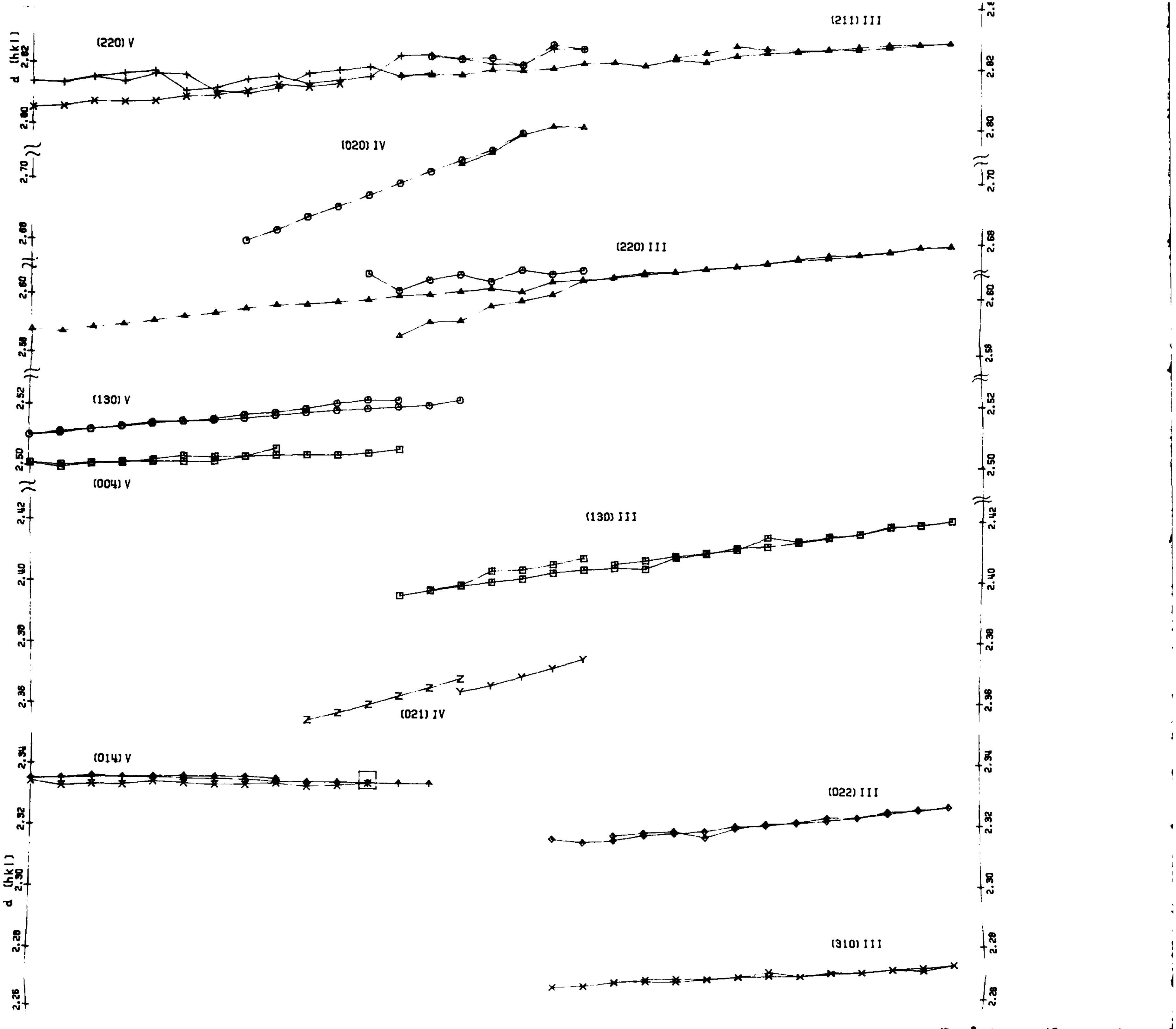


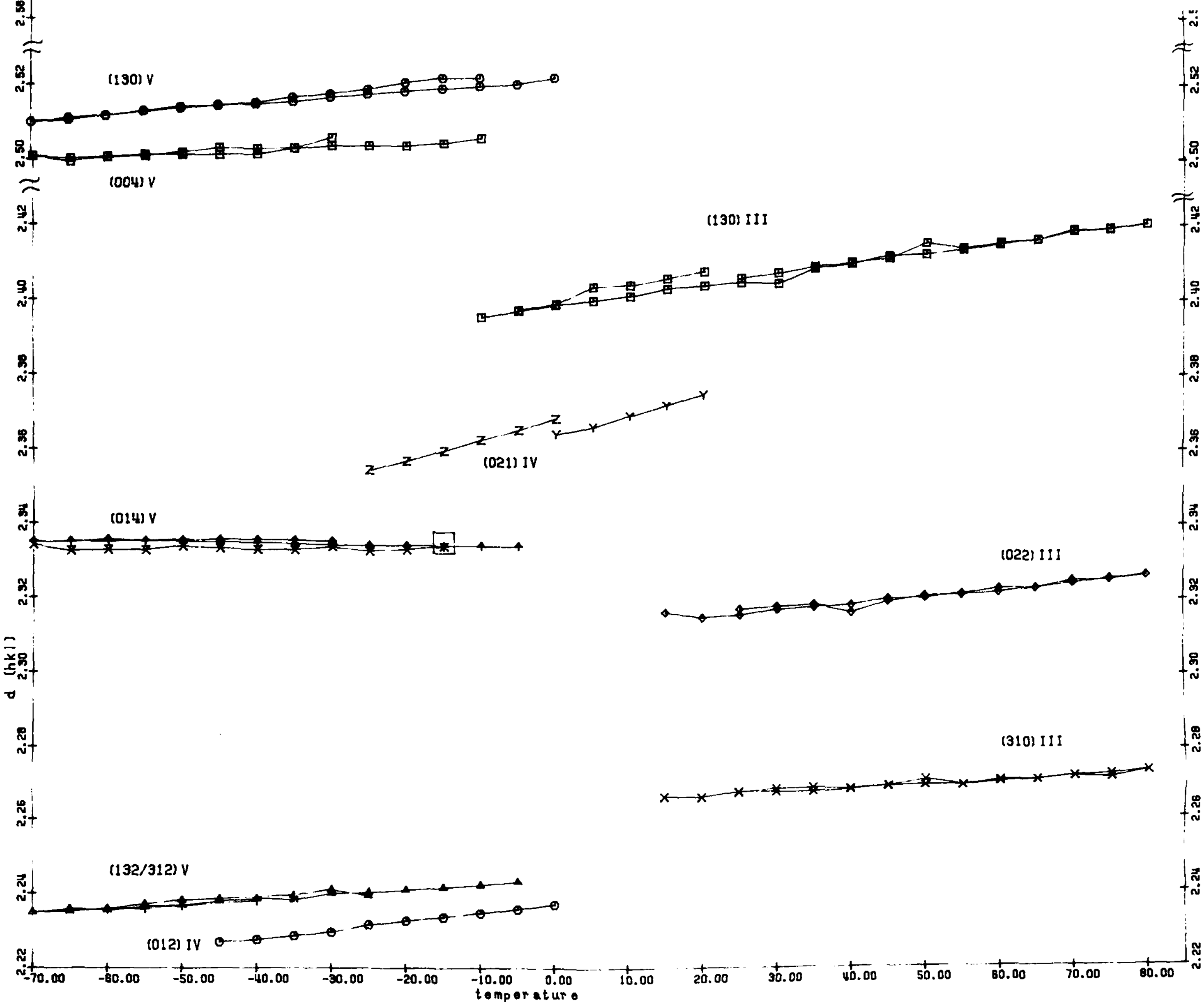


V (212)
V (220)
IV (111)
III (120)
III (210)
III (021)
III (111)
IV (110)
(211) III
d (hkl)
d (h'kl)



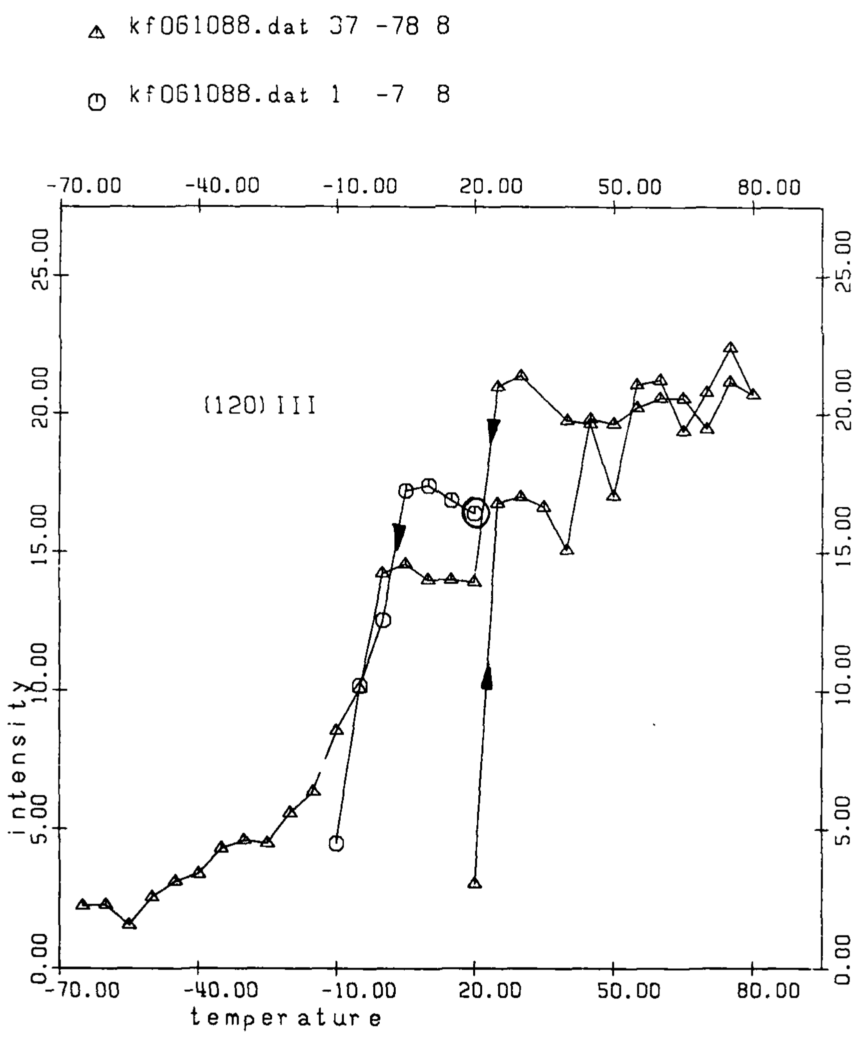
d (hkl)
(004) V
(130) V
(220) V
(212) V
(022) V
(020) IV
(111) V
(130) III
(220) III
(211) III
(021) III


(220) V
(211) III
(020) IV
(220) III
(130) V
(004) V
(130) III
(021) IV
(014) V
(022) III
(310) III
d (hkl)
2.82
2.80
2.70
2.68
2.60
2.58
2.52
2.50
2.42
2.40
2.38
2.36
2.34
2.32
2.30
2.28
2.26


(130) V
(004) V
(130) III
(021) IV
(014) V
(022) III
(310) III
(132/312) V
(012) IV
d (hkl)
2.22
2.24
2.26
2.28
2.30
2.32
2.34
2.36
2.38
2.40
2.42
2.50
2.52
-70.00
-80.00
-50.00
-40.00
-30.00
-20.00
-10.00
0.00
10.00
20.00
30.00
40.00
50.00
80.00
70.00
80.00
temperature

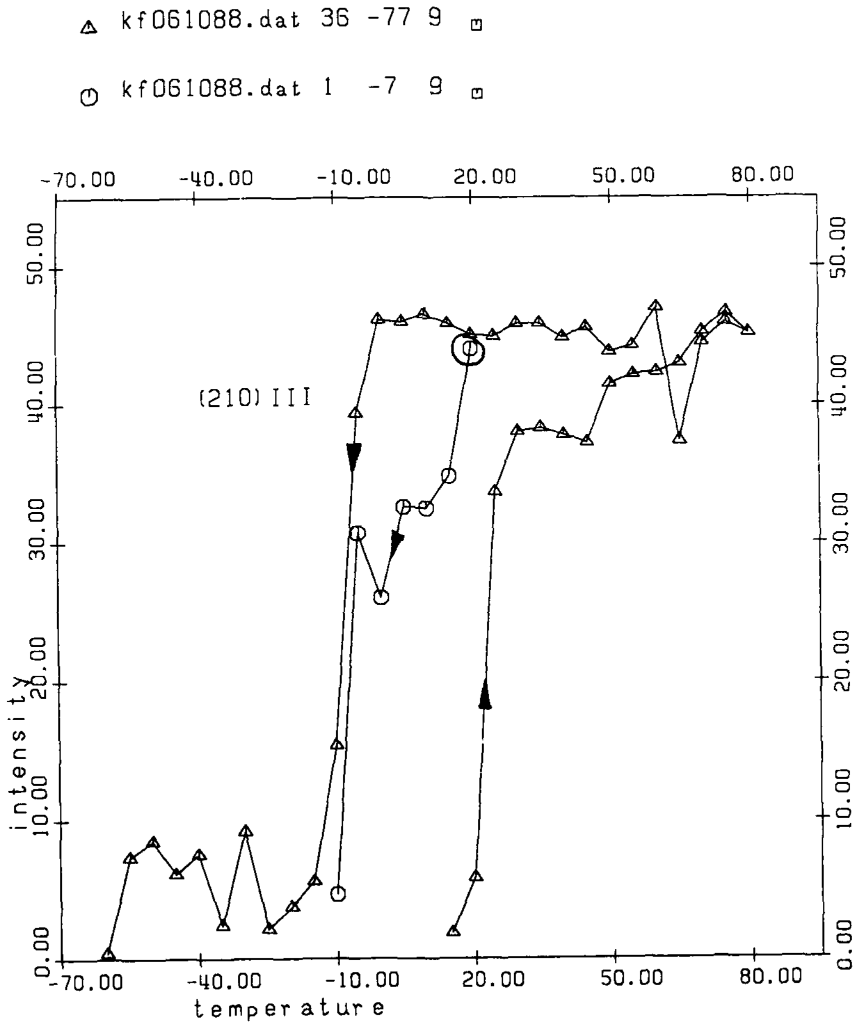
Intensities

KF061088
20/-70/80/-70



Intensities

KF061088
20/-70/80/-70



Intensities

KF061088
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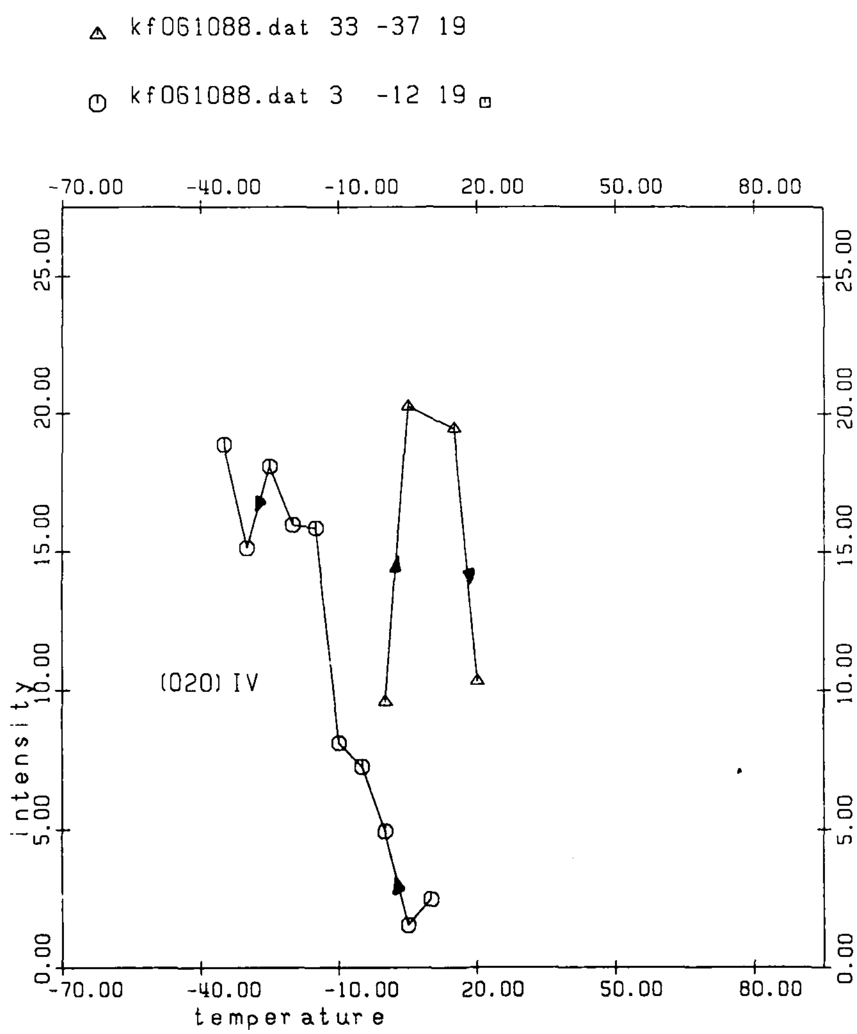

Intensities

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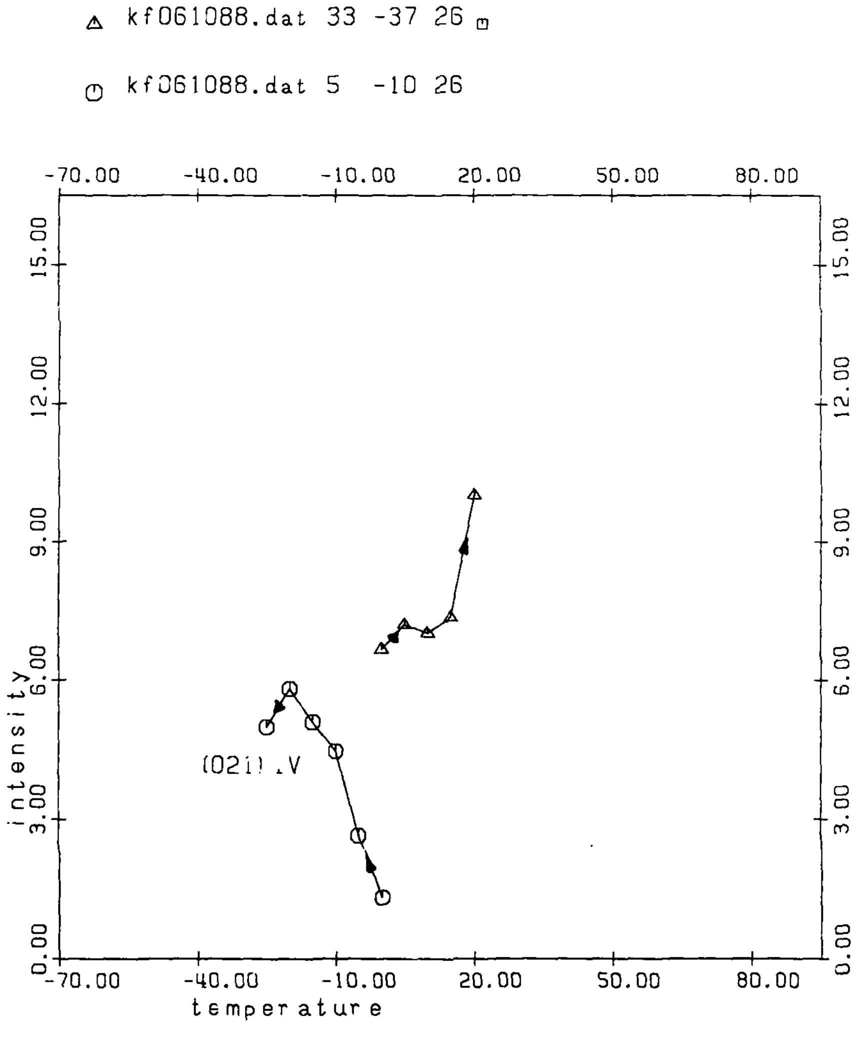
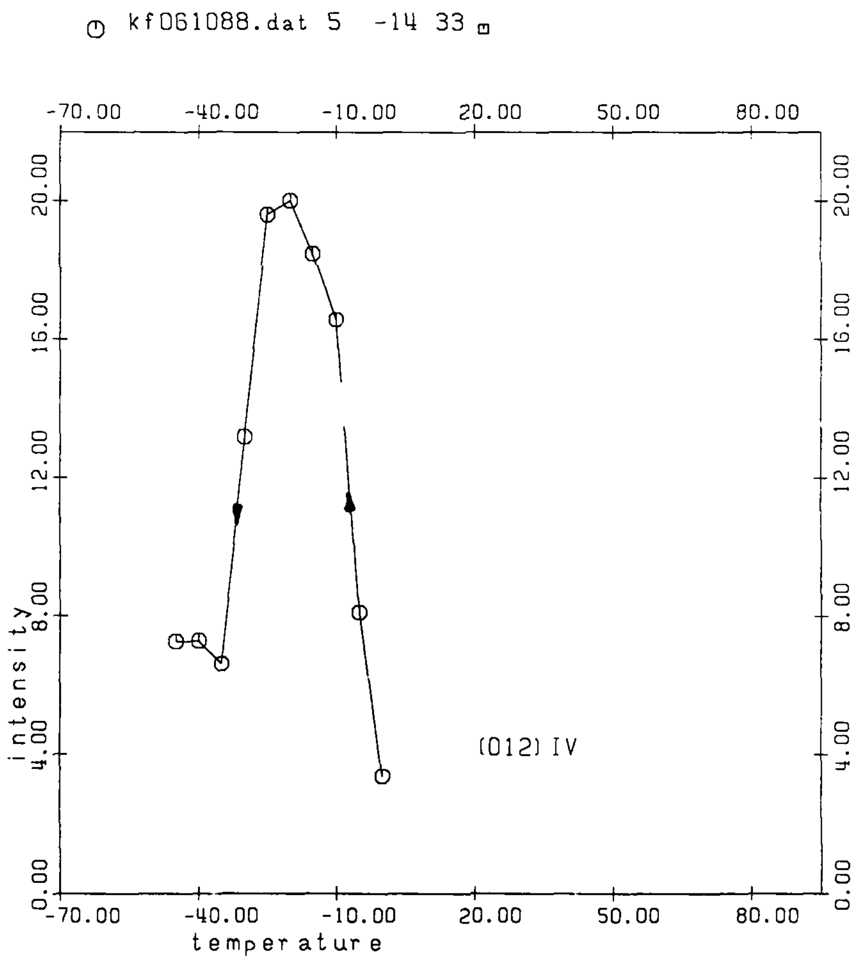


Intensities

KF061088

20/-70/80/-70



Intensities

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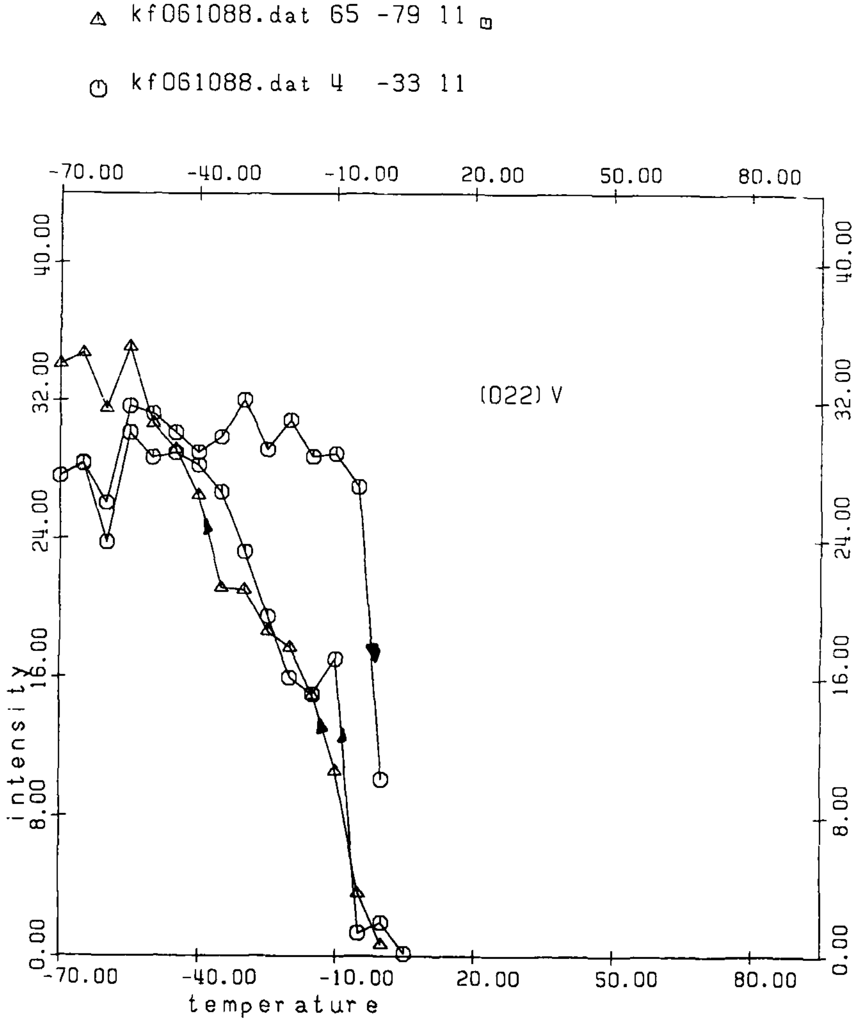

Intensities

KF061088
20/-70/80/-70